

2022151408

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MONTHLY PROGRESS REPORTS
Period Covered
August 1 - 31, 1981

Date Issued: September 15, 1981

2022151409

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2022151410

CHARGE NUMBER: Various

DATE: September 14, 1981

PROGRAM TITLE: Analytical Research

WRITTEN BY: G. Vilcins, G. H. Bokelman, R. H. Cox, R. E. Davis, W. R. Harvey,
J. O. Lephardt and D. C. Watson

I. TOBACCO ANALYSIS

A. Nitrite Monitor for SEL Denitrification Process

A monitor for nitrite consisting of peristaltic pumps for sampling and dilution and AA-I AutoAnalyzer equipment was installed in the Pilot Plant. Tank H was sampled continuously for 48 hours with good agreement with the Pilot Plant nitrite results. Work will continue to minimize the time for maintenance that the monitor requires.

B. Fractionation of Tobacco Polysaccharides

It appears that 0.10 M aq. KOH extracts tobacco almost as efficiently as does 0.50 M aq. KOH; however, 0.05 M aq. KOH is a much less efficient extraction solution. Experiments are in progress to determine if extraction with 0.10 M aq. KOH will minimize the apparent alkaline degradation of soluble polysaccharides.

C. Amino Acids by HPLC

An HPLC ion exchange method was implemented to monitor amino acids in the autogenous fermentation process due to constant breakdown of the Dionex Amino Acid Analyzer. Further studies are planned but results look very good.

D. Methylated Alditol Acetates

Methylated alditol acetates are presently being prepared from several different tobacco fractions. These derivatives will eventually be analyzed by G.C.-M.S. to give information on the linkage patterns of monosaccharides within the tobacco structural carbohydrates.

E. Four Component Analysis

Relative to an objective of alleviating the necessity for picking reconstituted material prior to performing ERA blend analysis, methods for performing four component analyses are being explored. To test the value of predetermining one component by an independent means and then fixing this value in the ERA procedure some simulations were performed. This procedure did not result in convergence to the theoretical values, however and other procedures will be explored.

II. FLAVOR ANALYSES

A. A program has been established with SEF and Flavor Development to study the analytical/sensory relationships of flavor components and formulations. Finished preblends will be prepared "out-of-spec" by purposely altering component concentrations in a sequential manner. Degree of difference values will then be compared, sensory versus analytical, to establish the basis for acceptance/rejection criteria.

B. Chromatographic profiles of production lots of preblends and of formulated components of preblends were compared to lab made references mixtures and to the individual components. The results show, among other things, that component interactions can be a major source of analytical (and presumably sensory) differences.

C. Methods are being developed for eventual application to cooked flavors and establishment of accept/reject criteria.

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III. SPECTROSCOPIC ANALYSIS

A. Mass Spectroscopy

A Porapak Q column (20' x 1/8" S.S.) was found to give good separation of O₂, N₂, H₂O, CO₂ and N₂O when used at subambient temperature with the MS as a detector. A sample of headspace air over Vat H in the Pilot Plant was taken and qualitative results are being generated from it. Quantitation will begin shortly when all reference standards have arrived.

B. FT-IR-EGA

Data acquisition for a study of Na/K nitrate/nitrite salts applied to cellulose has been completed. The data are presently being evaluated, particularly for the formation of oxides of nitrogen.

C. Infrared Spectroscopy

A method for the quantitative determination of polyvinyl acetate (PVA) on the seams of cigarette paper has been developed. The method involves extracting the PVA with chloroform. The absorbance of the carbonyl band of the PVA is obtained using the PE-283 infrared spectrophotometer. The amount of the PVA on the seam is determined by relating the absorbance of the unknown sample to the absorbances of the standard solutions of PVA.

Several attempts were also made to compare different adhesive samples by infrared transmission spectroscopy toward an objective of a quality control procedure for the adhesive. Transmission techniques were found to be unacceptable. An ATR technique was proposed and a Germanium ATR element (compatible with the aqueous base adhesives) was ordered to test this alternate approach.

IV. CHEMOMETRICS

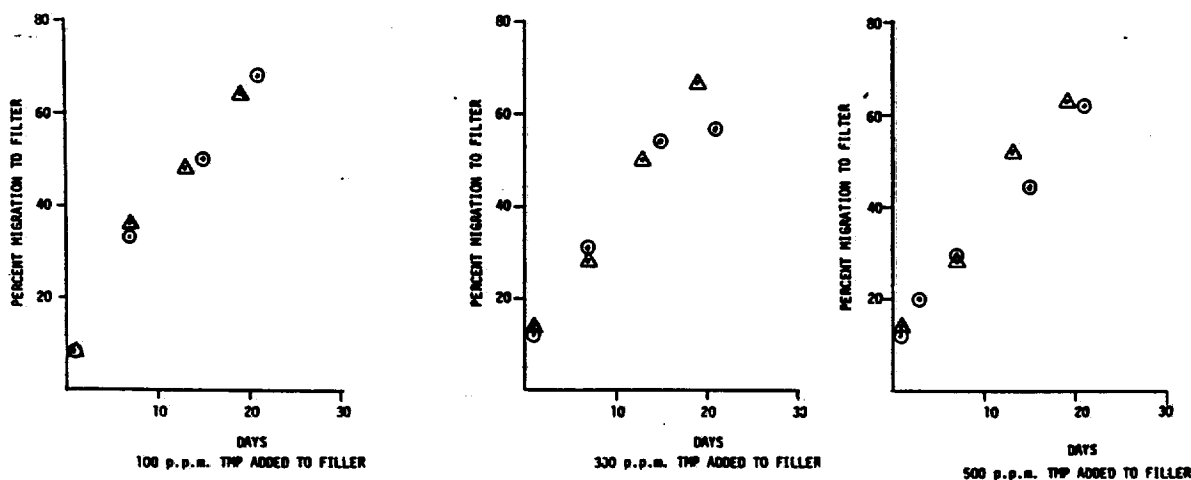
- A. The volatile phase (whole smoke) raw data that had been generated and stored on the Varian were recalculated and replotted with the baselines drawn to the best conditions. These profile reports were transmitted to the DEC 20/60. Due to data variation (aged/unstable gc column, hand-operated high pressure drop transfer valves) only 22 peaks were selected and digitized for Factor and Discriminant Analysis.

Guana Vilcin

2022151412

CHARGE NUMBER: 0108
PROJECT TITLE: Mechanism for Smoke Formation
PERIOD COVERED: August 1-31, 1981
PROJECT LEADER: R. A. Kornfeld
DATE OF REPORT: September 9, 1981

The study of tetramethylpyrazine (TMP) transfer from filler to filter in unsmoked cigarettes is nearly complete. As was mentioned last month, cigarettes stored at -25°C and -85°C showed no transfer. The data for the cigarettes stored at room temperature are shown graphically below. Target values for TMP application to filler were 100, 300, and 500 p.p.m. At each TMP filler loading cigarettes were made with filters containing 9% triacetin (O) or no triacetin (Δ). It is evident that transfer to the filter occurs rapidly (i.e. 1/3 of the TMP on the cigarette after seven days is in the filter). Also, it seems as if the presence of triacetin does not have an effect upon the transfer of TMP at room temperature. The filtration of TMP during the smoking process will be investigated using cigarettes whose filters are TMP-free (i.e. those stored at -25°C).^{1,2}



After considerable effort to reduce reactive surfaces in both pyrolytic and gas chromatographic apparatus, reproducible pyrograms are now being obtained with the nitrogen phosphorus detector. Research will now proceed on the pyrolysis of cooked flavors.^{3,4}

Several samples were analyzed using direct introduction mass spectrometry.⁴

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1. Two benzoyl derivatives of polyisoprenoids. (G.Chan)
2. Two TLC spots from the pyrolysis of a glyceryl cyclic carbonate designed to release phenol. (K. Podraza)
3. A synthetic byproduct in the reaction of glyceraldehyde with valspice chloroformate. The mass spectrum obtained was very similar to valspice carbonate. (K. Podraza)

Ether extracts of two cooked flavor samples (submitted by D. Douglas) were analyzed using capillary GC/MS. In addition to a large number of pyrazine derivatives, several aliphatic acids were detected and identified.*

The following memoranda and reports were written this month:

1. "GC/MS Analysis of Glue Samples G3910, G3802 and G3969" memo to Dr. Tom Van Auken from J. D. Naworal, August 19, 1981.
2. "GC/MS Multiple Ion Detection Analysis of Nicotine in Blood" memo to Dr. Vic DeNoble from J. D. Naworal, August 27, 1981.
3. "WS-14 (N-t-butyl-p-menthane-3-carboxamide) as an Additive to Cigarette Paper", Special Report by A. F. Frisch and R. A. Kornfeld, Accession No. 81-207, August 17, 1981.

References

1. L. Brown
2. C. Connolly
3. J. Kang
4. J. Naworal

Rich Kornfeld

2022151414

CHARGE NUMBER: 0307
PROJECT TITLE: Cigarette and Tobacco Measurement Methods
PROJECT LEADER: C. L. Irving
PERIOD COVERED: August, 1981
DATE OF REPORT: September 9, 1981

I. Moisture Measurement

Analysis of data collected on burley strip samples with and without OTM showed a range of correlation coefficients from 0.78 to 0.90 between microwave attenuation and tobacco moisture (OV or GC water). The samples were tested at approximately one half hour intervals as they equilibrated from production line ($\approx 35\%$ OV for burley with OTM, ≈ 24 for burley control) to laboratory conditions. Considerable scatter was evident in the data because of the nonuniform moisture of the fresh samples. An accuracy of moisture prediction for the microwave unit could not be calculated because of the nonuniform moistures, but the prediction equations can be used to obtain estimates of sample moisture.

A trip was made to Steinbrecher Corporation to verify that work on the automatic microwave moisture meters is progressing on schedule. The review of the work to date shows most items to be on schedule with the exception of the X-band converter. The time lost on the X-band converter design was spent to linearize the variable attenuator over the working frequency range. The remainder of the job is progressing well and the critical design review is tentatively scheduled for mid October.

II. Measurements Development

Testing of the RoTap sieve tester continued with tobacco conditioned to high moisture levels ($\approx 22\%$ OV) in an effort to prevent tipping and therefore reduce noise in the test. The results of the experiment, however, showed the data from the high moisture tobacco to be just as noisy as the normal moisture samples. No further testing of the RoTap sieve tester is planned unless the evaluation of the PM sieve tester shows significantly worse results. A standard PM sieve tester was received and is being evaluated for efficiency of particle size segregation using the two image analysis systems as standards.

A test was completed to evaluate a ball mill, used in conjunction with a sieve test, as a device for measuring tobacco friability. For the test, Marlboro filler was tested in the ball mill with two levels of tobacco loading, at two ball loadings with two different densities of balls, and at three different running times. Mr. J. Tindall's analysis of the results, using sieve fractions as a friability indicator, showed only ball load and running time to be significant variables. His analysis showed the results did not fit a predictable pattern but were repeatable. Since the results are repeatable an additional experiment is planned that will determine if a difference in friability can be measured for six different tobacco types. This experiment will be done at only one ball loading and running time.

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An experiment was also completed using a Fitz mill, in conjunction with a sieve tester, as an indicator for tobacco friability. The Fitz mill is a rotating blade device that forces the material under test through a screen. For this test, tobacco was degraded using both shear and impact blades at 2500 and 950 RPM with four screen mesh sizes. The results showed the amount of material retained on a 35 mesh screen on the RoTap sieve tester increased as the mesh size of the screen on the RoTap was reduced. The results also showed impact to be more degrading than shear at the high speed, but little difference was observed at the low speed. An additional test is planned with six tobacco types at a single operating condition to determine if differences between tobacco types can be measured.

III. Cylinder Volume

The semi-automatic CV tester was received from Engineering Services and is being checked out to be sure it meets mechanical specifications. Testing with tobacco is planned to begin by September 14. Data from the tests with tobacco will be analysed to compare the results from the semi-automatic unit with standard cylinders at five minutes and to investigate the possibility of reducing the test time.

Christopher L. Ling

2022151416

CHARGE NUMBER: 1005
PROJECT TITLE: Improved Semiworks Operations
PROJECT LEADER: J. F. Sherwood
PERIOD COVERED: August, 1981
DATE OF REPORT: September 9, 1981

I. Primary Processing

A. Export Filler Shipment

A test was completed, at the request of Manufacturing, to determine the cause of OV changes in bales of export filler during shipment. The results of tests, in which bales were placed in the jungle and desert rooms, showed significant filler OV changes were caused by small tears in the polyethylene bags covering the bales rather than moisture diffusion through the polyethylene. It was recommended that care be taken to avoid tearing the bags and that the bales be double bagged.

B. WS Extraction

A feasibility study was completed to investigate the possibility of extracting Northwind factory by-products with liquid CO₂ to recover the WS and allow the by-products to be used in sheet plants without danger of contaminating reconstituted tobacco with WS. Test results showed that in a single batch extraction, the CO₂ extracted approximately 90% of the WS from Northwind class tobacco without removing significant amounts of other tobacco components. A cost estimate for a potential production scale extraction process will be prepared to determine if it is economically feasible to extract by-products with liquid CO₂.

C. Burley Equilibration Silo Depth

At the request of Manufacturing, an experiment was run to determine if cased DBC burley strip could be stored in an equilibration silo at a depth of 8 feet, instead of the normal 6 feet, without adversely affecting cut filler CV or sieve fractions. Increasing the silo depth to 8 feet would allow increased capacity without requiring additional floor space. In this test, the bottom two feet in the silos were simulated by placing cased burley strip exiting the P&S apron dryer in the Pilot Primary in saratogas and compressing the tobacco with the appropriate weights to obtain the pressures that would occur in the bottom two feet of silos with tobacco depths of 6 and 8 feet. After storage in the saratogas for 45 minutes (the silo hold time), the burley strip was processed through the remainder of the Pilot Primary. The results of tests on four lots of DBC burley strip showed no significant differences in cut filler CV or sieve fractions between the samples stored at simulated silo depths of 6 and 8 feet.

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II. New Semiworks

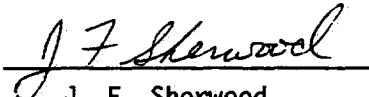
Startup activities in the Primary Pilot Plant in the new Semiworks are continuing with tobacco being fed through the hogshead splitter and strip opening lines to check for proper equipment operation and for choke points.

Installation of the flooring in the Make/Pack area was completed and delivery of rebuilt equipment from the York facility should begin shortly.

III. Cigarette Making and Packing

Cigarette Firmness Specification

A program was initiated under the direction of Operations Services to ensure that all Philip Morris brands have an average 24 hour pack cigarette firmness of 30 mmx10. In this program, the as-is firmness levels of production cigarettes are being measured in the Materials Evaluation Facility after aging in packs 24 hours. Initial monitoring of three brands produced Aug. 11 through Sept. 9 in the MC was completed. The results showed Marlboro KS to have an average firmness of 29.6 ± 0.7 mmx10 (95% confidence interval on the mean), Marlboro LS to have an average firmness of 32.2 ± 0.4 mmx10, and Marlboro 100's S.P. to have an average firmness of 28.7 ± 0.6 mmx10. As cigarette weight specifications are changed to adjust average firmness, further firmness tests will be made to confirm that the firmness specification is met


J. F. Sherwood

JFS:jn

2022151418

CHARGE NUMBER: 1101
PROGRAM TITLE: ENTOMOLOGICAL RESEARCH
PERIOD COVERED: August 1-31, 1981
PROJECT LEADER: M. A. Manzelli
DATE OF REPORT: September 8, 1981

I. INSECT GROWTH REGULATOR

A. Stemmary 5 ppm KABAT® Study

Bright strips treated nine months previously (November, 1980) at a 5 ppm methoprene level were assayed both chemically and biologically. Results were as follows:¹

<u>Tobacco</u>	<u>Methoprene, ppm</u>	<u>Beetle Emergence</u>
Control	0	39%*
Treated	5	0
Treated	5	0
Treated	6	0
Treated	5	0

*Adult beetles still emerging.

B. Methoprene Residue in Cigarettes

A study has been initiated to determine the methoprene residue in cigarettes made from tobacco treated with 5 ppm methoprene. Tobacco from the 1980 stemmary treatments and from the 1981 season have been employed to make the blends.²

C. Maury Street Warehouses/KABAT® Study

Twenty one weeks of trap counting has given the following data:³

<u>Treatment</u>	<u>Beetles per Warehouse per week</u>
None (Control)	1700
Standard (PH ₃ and DDVP)*	17
KABAT®**	17

*One PH₃ treatment plus 105 to 147 DDVP treatments.

**One KABAT® treatment.

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II. CIGARETTE BEETLE PHYSIOLOGICAL STUDIES

Field studies (warehouse) with the PM synthesized sex pheromone are underway. The Japanese sex pheromone product (Serricornin) is being tested concurrently.⁴

III. ASSISTANCE TO OTHERSA. Efficacy Study of Automatic DDVP Dispensers

At the request of the Leaf Department, the automatic DDVP dispensers in four of our storage locations were tested for adult beetle kill (Park 500, Alleghany, Star, and Maury Street). The mortality data obtained showed that the devices were effective.⁵

B. Phytosanitary Certification Efficacy Tests

The 20th Street Guardite unit and Thermovac Chambers were tested for beetle kill efficacy as part of the phytosanitary certification requirements. All exposed beetle larvae and adults were killed.⁶

IV. REFERENCES

1. Lehman, R. M. Notebook No. 7235, pp. 173-4.
2. Lehman, R. M. Notebook No. 7235, p. 175.
3. Minor, M. F. Notebook No. 7197, p. 107.
4. Long, J. S. Notebook No. 7283, pp. 167-8.
5. Long, J. S. Memo to H. L. Wright. 1981 August 25.
6. Lehman, R. M. Notebook No. 7235, p. 176.

nwp

M. M. Wright

2022151420

CHARGE NUMBER : 1307

PROGRAM TITLE : Reconstituted Tobacco Development

PERIOD COVERED: August 7-September 8, 1981

PROJECT LEADER: G. Gellatly

I. RL PROCESS

Preliminary pilot plant trials were run at 40 fpm using the recently installed coater which demonstrated the feasibility of sizing one side of RL sheet and coating the other with a homogenate of size and CT. Some minor mechanical modifications are required before the product evaluation program is begun. During these trials a finished sheet weight (17.2 gm/ft^2) of up to three times the base weight was demonstrated. This is equivalent to applying all class tobaccos currently in the feedstock as an homogenate in CEL to an all stem base web.

II. RL PRODUCT

Park 500 trials to evaluate the effect of stem content on RL sheet quality, production rate and yield were completed and a report will be issued in October.

Park 500 trials to quantify the effect of improved refining on RL sheet quality and production rate will begin in September and are expected to be completed by the end of the year. New refiner plate designs which have been shown to improve sheet quality in the pilot plant will be evaluated.

A promising means of quantifying sheet formation by scanning the sheet with a photo electric cell and measuring the amplitude of the variation on a strip chart is being developed. Previously, sheet formation has largely been a visual subjective judgement.

III. CT APPLICATION TO TOBACCO MATERIALS

Several MC trials were run which demonstrated that 7.5% homogenized CT could be applied to burley strip with a reduced water/casing ratio (0.25/1; original formulation 1/1), which could be dried in the P&S driers to specification OV (21+1%).¹ The adhesion of the CT and the filling power of this CT burley product using the new formula is being determined.

Sand mills from three different manufacturers have been rented and will be evaluated in R&D for homogenization efficiency and durability to make a choice of homogenization efficiency and durability to make a choice of homogenization equipment for an MC installation before November. One of these sand mills has demonstrated 100% partical size reduction below 5μ in the laboratories of Tennessee Eastman.

The equipment for application of homogenized CT to cut stem was tested in R&D and has been shipped to Louisville Factory for production trials. These trials will evaluate the application of CT/casing homogenate in the superwetting step followed by expansion in the ES tower. Demonstration trials are expected to begin during the week of 09/21/81.

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CT has been successfully applied to whole cut filler (2% CT) as 17% homogenate in water without the filler balling up. A CV increase of 1.7 units was obtained for both the test product and control (water reordered). Cigarettes have been made to evaluate this product subjectively.

IV. RCB

Alternative means of evaluating the effect of microwave treatment of RCB were considered and equipment is being rented from Cober, Inc. because this was the most expedient way to complete this evaluation before the BL Pilot Plant is dismantled in December 1981.

A promising means of separating charcoal from ripper shorts is being evaluated using a Fluidized Bed Reclaimer made by SWECO. A tobacco recovery >80% (containing <0.4% charcoal) was indicated from feedstock containing ~3% charcoal. A discard product containing ~30% charcoal would result from this separation.

VII. REFERENCES

1. "M/C Primary Trial of STP Application to Burley" to C. G. Bates from C. H. O'Donohue dated 07/01/81, 07/16/81, 08/11/81, 08/13/81, 08/19/81, 08/25/81, 08/27/81, 08/20/81, 08/31/81 and 09/01/81.



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CHARGE NUMBER : 1503

PROGRAM TITLE : Modified Smoking Materials

PERIOD COVERED: August 1-31, 1981

PROJECT LEADER: G. D. Keritsis

I. RCB STUDIES (J. W. Leik)^{1,2}

It was previously reported that the homogenization of production RCB slurries with a Gaulin homogenizer increases the tensile of the laboratory cast/dried RCB sheets by 35-40%, and that the addition of 2% NaCMC 7HF (DWB) to the homogenized slurry increases the sheet tensile by an additional 20-40%.¹ Hence, the study was repeated with stem free tobacco dust slurries and with slurries containing varying amounts of bright stems in place of burley stems.

The laboratory data indicates that the control sheet physicals (tensile, TEA, etc.) were matched by adding 3% of certain gums to stem free tobacco dust. The addition of 3% α -cellulose or a minimum amount of bright stems seemed to have little effect on tensile.² A few of these formulations are now being repeated prior to making any runs at the BL Pilot Plant.

II. WATER EXPANSION (H. H. Sun)^{3,4}

The WET studies with bright, burley, Oriental and MF fillers were continued in an effort to optimize the process. The studies indicate that a high degree of expansion is achieved when the tobacco filler, regardless of feed OV, reaches a "dry state" of less than 10% OV and preferably less than 6% OV for a duration of less than four seconds of exposure in the "dry state" at a tower temperature of >450°F.

The evaluation is continuing of the Westab WET (07/19/81 test) using uncased DBC bright. The test results indicate that this filler could be post-cased with a minimal CV loss of about 2-3 CV units (61 cc/10 g vs 59 cc/10 g). This technique was then used to produce blended cigarettes for an evaluation which is currently underway.

III. FUNDAMENTAL STUDIES

A. Denitration of Tobacco by Chemical Means (S. E. Wreen)⁵

Efforts are being made to scale up the process of denitrating tobacco with gaseous formic acid and developing means of recovering excess formic acid.

B. Selective Filtration of Smoke (N. B. Rainer)⁶

It has been found that certain types of granular carbons can adsorb small quantities of metallo-organic compounds which are either applied from solution to the granules or synthesized directly in an adsorbed state on the granules. Specially treated carbons activated in such a manner and utilized in PSP filters have been found to adsorb 80% of NO from cigarette smoke. Aging tests are in progress. Also optimization studies continue on metallo-organic compounds and the manner of granule treatment.

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In another study with permanganate filter granules it was learned that the optimum space fill level in a PSP filter is 100% for filter granules having 30/80 mesh size. When the granules are of a finer mesh size, such as 60/80, the optimum space fill is at about 80%.

C. Cellulase Treated Classed Tobacco (H. H. Sun, M. Shulleeta)^{7,8}

Earlier studies have shown that cellulase (*Trichoderma viride*) can degrade RKS into very fine, soft fibers which can easily be homogenized with a Waring blender. It was then thought that such a treatment could be applied to OTM to soften and degrade CT particles for an easier application to strip or RL. Such treatments were studied, and it was found that the treatment of a homogenized CT slurry with 4% (DWB) cellulase reduced the slurry viscosity to one-sixth of the control and that the treatment with 1% (DWB) cellulase reduced the homogenized slurry viscosity by 50%. The evaluation of these treatments will continue with Project 1307 personnel.

IV. REFERENCES

1. J. W. Leik Notebook Numbers 7395 and 7644.
2. J. W. Leik Notebook Number 7644, page 12.
3. H. H. Sun Notebook Number 7379, pages 147-152.
4. M. Shulleeta Notebook Number 7290, pages 109-125.
5. S. E. Wrenn Notebook Number 7621, pages 61, 64-70.
6. N. B. Rainer Notebook Number 7278, page 200.
7. H. H. Sun Notebook Number 7379, page 153.
8. M. Shulleeta Notebook Number 7290, pages 126-131.

B.D. Keith

/deb

2022151424

CHARGE NUMBER: 1600
PROJECT TITLE: Smoker Psychology
PERIOD COVERED: August 1 - 31, 1981
PROJECT LEADER: W. L. Dunn
DATE OF REPORT: September 9, 1981

The Effects of Cigarette Smoking on Brainstem Auditory Evoked Potentials(F. Gullotta)

Initial data analyses have been completed and a memo is currently being written. The data suggest the possibility that smoking influences structures in the vicinity of the lower mid-brain. However, additional research would have to be done in order to confirm these findings.

Duration of Effect Study (F. Gullotta)

Thirty seven tests have thus far been completed on ten individuals. It is estimated that the study will be finished in November.

Perception and Pattern Reversal Evoked Potentials: Smoking Effects (F. Gullotta)

The pilot phase of this study is nearing completion and some preliminary analyses are under way. A decision on whether to undertake a full-scale investigation will be possible by early October.

Changes in Smoke Inhalation Patterns as a Function of Changes in Nicotine Delivery (Jan Jones)

Subject recruitment, data collection and data analysis continue.

Inhalation Patterns on Market Brands and their "Light" Versions (Jan Jones)

Data collection is underway on a study observing inhalation behavior as a subject smokes a current market brand of cigarette, then switches to the "light" brand of the same name.

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Study of Psychosocial Factors Influencing Smoking Behavior
(S. Dunn)

Computer entry of the data for further in-house analysis nears completion. Our next task will be to examine the data for variables that discriminate between the high and low delivery brand smokers.

Study of the Perception of the Social Desirability of Smoking
(S. Dunn)

A scoring key for the Adjective Checklist has been designed and hand scoring of these questionnaires is in progress.

Differences in Nicotine Intake Between High and Low Delivery Smokers (S. Dunn)

Twenty of the high delivery smokers and twenty of the low delivery smokers who were participants in the Psychosocial Project have saved the butts from all cigarettes smoked for two consecutive days. Each day's supply of butts was put in an aluminum foil pouch and returned to us in pre-postage paid mailers. We are preparing the butts for nicotine analysis by the R&D Analytical Division.

Influence of Situational Events on Smoking Behavior (S. Dunn)

We have decided to obtain an additional behavioral measure in this study. We plan to obtain a measure of each person's need for stimulation. We believe those smokers who are most motivated by situational events to light a cigarette, will be those people who are most in need of all kinds of environmental stimulation.

Consultant Search (S. Dunn)

Three candidates have been recommended for the program consultant position. We are in the process of making a final decision.

Firmness and Acceptability (F. Ryan)

A panel of 40 R&D employees has rated the acceptability of 13 different experimental cigarettes which differed in firmness.

Panelist responses indicate that there are two types of people in the R&D population: one which says "the firmer the better", and one which says something else, such as "not so firm", "soft", or "reasonably firm, but not too firm".

The latter panelists downgrade very firm cigarettes severely. Given the presence of the two types of raters and the source of the panel it is impossible to make unqualified statements about acceptability. However, the data available imply that cigarettes with compacimetric numbers between 31.5 and 29.0 will be acceptable to most people. The "firmer the better" people don't like cigarettes with compacimetric numbers higher than 31.5, and the

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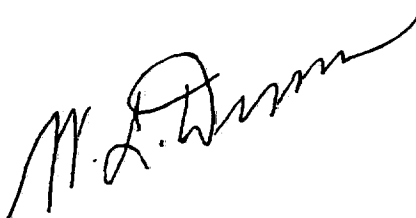
other panelists downgrade severely beyond 29.0.

No apparent differences can be attributed to brand usually smoked, gender, etc.

Firmness and Acceptability (F. Ryan)

A large group of POL panelists is ranking the importance of a variety of lit and unlit cigarette characteristics. Ballots are due by September 11, and about 200 usable ballots have been received as of September 8, 1981.

/iw

A handwritten signature in cursive script, appearing to read "M. L. Ryan", is written diagonally across the page.

2022151427

PROJECT CHARGE: 1702

PROJECT TITLE: FILTRATION PHYSICS

PROJECT LEADER: R. W. Dwyer

PERIOD COVERED: August 1 - 31, 1981

DATE OF REPORT: September 10, 1981

A study has been completed on the effects of basic design parameters of CA tow filters on their porosities, pressure drops, and filtration efficiencies. It has been found that each of these filter characteristics can be predicted from the geometry of the filter and the denier of the tow. The effects of condensation on filtration efficiency are also being studied. For unventilated filters, it has been found that the contribution of condensation increases the measured efficiency exponentially as the distance between the coal and the filter decreases. The role of filter ventilation on this process is now under study. A protocol has been developed for characterizing novel filters. This involves determining the filtration efficiency per unit pressure drop of the material, as well as its pressure drop-flow behavior over the flow range of 0 to 2000 cc/min. Additionally, photomicrographs of the filter materials are made in order to determine their size and shape factors. This analysis shows that, at the same pressure drop, the trend in TPM efficiencies for a variety of filter materials is:

VAT > CONVENTIONAL CA TOW = FOAM CA > POLYPROPYLENE TOW >> COD

This observations suggests that the COD filter would make an excellent candidate for a high RTD-low efficiency filter (1,2).

Cigarettes with two different filter designs were submitted to the Flavor Development Panel for comparative evaluation. Both samples delivered 5 mg of TPM at RTD's of 4.1 in (H₂O) and ventilation levels of 51%. One sample was comprised of a 20mm section of 2.1/60K tow with a 10mm polyethylene recess at the mouth end. The vents in this filter were located at the recess-filter junction. The second sample had a 30mm filter of 2.5/32K tow with the vents located 15mm from the mouth end. Both filters were attached to MUL rods. The Panel found the recessed filter to give a harsher, hotter smoke with a plastic off-taste. The Panel also smoked the 2.5/32K filters paired with production MUL's. They found the 2.5/32K version to give "increased impact and response, increased smoke volume, hotter, drier, and a response similar to Marlboro." These filters are being applied to Cambridge to see if these flavor notes persist at this lower delivery level (3).

The Multiple Wavelength Light Extinction Photometer (MWLEP) has been refined. The photodetectors were calibrated and found linear over the extinction region of interest, and the cell system was redesigned. A prototype instrument is being designed which can incorporate more detectors than

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the four used on our current model (4).

Calibrations of the MWLEP and cascade impactors are continuing. Mono-disperse aerosols of DBP have been produced over the range of 0.26 to 0.61 μ m. The agreement between the MWLEP and the Light Scattering Photometer aerosol size measurements is good. Additional runs at larger particle sizes are being made (5,6).

Work has continued on the calculation of aerosol coagulation coefficients. Some unexpected results have been obtained. These data are being analyzed to determine whether they are real anomalies or a consequence of the numerical scheme used to generate them (6).

An evaluation of the literature on the physical properties of sidestream smoke has been completed, and a seminar on this topic was given. A report is being prepared (7,8).

REFERENCES

- 1) S. G. Abel
- 2) R. W. Dwyer
- 3) M. L. Fleming
- 4) R. M. Creamer
- 5) D. M. Puzio
- 6) K. A. Cox
- 7) D. D. McRae
- 8) D. D. McRae: "The Physical Properties of Side Stream Smoke," presented at the P.M. Side Stream Symposium, August 27, 1981.

RW Dwyer

/ev

2022151429

PROJECT CHARGE: 1703

PROJECT TITLE: CIGARETTE MAKING TECHNOLOGY

PROJECT LEADER: A. Robinson

PERIOD COVERED: August 1 - 31, 1981

DATE OF REPORT: September 10, 1981

ADHESIVE PROPERTIES (T. E. Majewski)

The adhesive GC/Headspace analysis work has continued. The analysis technique was evaluated as a possible tool to monitor PVAc-based side-seam adhesives for batch-to-batch chemical consistency. The technique was evaluated on two Polymer Industries adhesives from different lots. There were no obvious dissimilarities in the adhesives chromatograms.

Infra-red analysis was used to determine if certain compounds identified in adhesives' headspace could also be identified by IR.

It was found that the IR band at 710 cm^{-1} in the plasticizer Benzoflex 9-88 (dipropylene glycol dibenzoate) spectrum was also prominent in an Ajax 563-8 sample spectrum.

ADHESIVE SURFACE BEHAVIOR STUDIES (R. G. Armstrong)

Contact angle and surface tension measurements were made on mechanically worked adhesive samples from the two Ajax 563-8 products which caused production problems during the week of June 29.

A value for an adhesion tension parameter was calculated from the measurements results. The magnitude of the parameter indicates the relative affinity (i.e., spreadability) an adhesive has for a specific substrate.

The results are as follows:

<u>Working Speed</u> <u>(rpm)</u>	<u>New Plasticizer</u> <u>dyne/cm</u>	<u>Old Plasticizer</u> <u>dyne/cm</u>
0	8.97	8.55
3,500	5.07	14.02
6,800	4.70	12.69
10,000	9.86	11.59
14,000	12.31	11.23
17,000	9.91	13.07
20,000	12.60	13.38
23,000	13.64	13.27

The new plasticizer adhesive was observed as having a greater propensity to buildup on the production machinery.

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RHEOLOGY (C. B. Hoelzel)

Homogenized OTM samples (previously called STP) continue to be received from the Tobacco Products Standards Group for rheological examination. Rheological curves were obtained for samples selected to represent variations in density and solids content.

NEW ADHESIVE SCREENING TESTS (C. B. Hoelzel and A. Robinson)

An incubator oven was adapted to serve as a humidity chamber for the preliminary adhesive moisture resistance test measurements.

The initial measurements were made on lap-seam samples at 54°C/75% RH for 31 hours and 25°C/100% RH for 44 hours. Under neither condition was evidence of separation noted. Further work is in progress.

In consideration of the results from the adhesive thermal analysis study⁽¹⁾, a thermomechanical analyzer (TMA) was introduced to Manufacturing as a possible adhesive physical property screening tool⁽²⁾.

REFERENCES

- 1) A. Robinson, "The Effect of Plasticizer Change in Ajax 563-8 Products," memo to G. M. Crowe, 8/21/81.
- 2) A. Robinson, "An Instrument for Screening Incoming Adhesives," memo to R. M. Gilman, 8/21/81.

A. Robinson

/ev

2022151431

PROJECT CHARGE: 1706

PROJECT TITLE: TOBACCO PHYSICS

PROJECT LEADER: D. B. Losee

PERIOD COVERED: August 1 - 31, 1981

DATE OF REPORT: September 9, 1981

APPLICATION OF TECHNOLOGY (1,2,3,4,5)

The initial phase of research has been completed on the rate of heat transfer from a smoldering cigarette to an object in intimate contact with this smoldering cigarette. Regression analysis of the data on a series of commercial brands indicates a significant correlation between several of the physical parameters of the cigarette. Among the parameters examined the most important is the static burn time. Not surprisingly, there is an increase in the rate of heat transfer resulting from a decrease in burn time. The next most important parameter is the rod density which gave a decrease in the rate of heat transfer with an increase in density. Furthermore, an increase in cigarette circumference could be correlated with an increase in the rate of heat transfer. The fourth most important parameter was the percentage of expanded lamina present in the blend. An increase in this percentage increased the rate of heat transfer. The least important parameters were the paper additive level, paper porosity and the percentage of stems in the cigarette blend. A report describing this research is in preparation.

Work continues on the tower modelling effort with the inclusion of loading effects. Through the cooperation of Ms. Beth Donenfeld data and reports on the heat transfer and particle velocities for 4", 6", and 8" towers were obtained.

Recommendations were made to Ms. Carol Wicks concerning an instrumental method for distinguishing normal DIET from toasted DIET. Initially, the rental of a Hunter Lab tristimulus colorimeter was proposed for the off-line phase of the program.

N-IR diffuse reflectance data collection has begun on the seven component blend analysis.

Heat treatment runs were completed in the Parr Bomb on bright tobacco at 93°C for 48 hours in air and N₂ (< ½% O₂). Preliminary examination of the data shows little effect of the atmosphere in the sealed container on the major evolved species. The only significant difference thus far noted is the conversion of ambient O₂ into CO₂.

MODELS FOR COMBUSTION/PYROLYSIS (6,7,4,5,8)

O₂-chemisorption on 750°C pyrolysis tobacco residue was studied at 25°, 35°, 50°, 65°, 100°, 125°, and 150°C. The results indicate an increase in

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the amount of chemisorbed O_2 with an increase in temperature, while the kinetics can be described by the Elovich equation. Interestingly, from data obtained at $100^\circ C$ and above there is an indication of the presence of at least two types of chemisorption sites on the tobacco residue. In order to further quantify these results improvements in instrumental stability are being pursued.

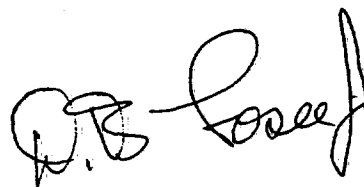
Nicotine, evolved as a function of temperature fraction, decreased in air by approximately 30% compared with that amount evolved in N_2 . However, the peak temperature and the temperature profile remained the same.

A report on the coordinated pyrolysis mass balance study conducted as a function of $25^\circ C$ temperature intervals is now in preparation.

Data collection on the isothermal effects of over 80 inorganic salts added to cellulose has been completed. Data have been plotted and are now being interpreted.

REFERENCES

- 1) C. O. Tiller, Notebook 7603.
- 2) D. L. Simpson, Notebook 7585.
- 3) P. A. Wilson, Notebook 7365.
- 4) B. E. Waymack, Notebook 7562.
- 5) K. R. Squire, Notebook 7440.
- 6) J. F. Bebbs, Notebook 7449.
- 7) P. E. Phillips, Notebook 7587.
- 8) A. Lewis, Notebook 7666.



/ev

2022151433

CHANGE NUMBER: 1708
PROJECT TITLE: Physical and Chemical Properties of Tobacco
PROJECT LEADER: B. C. LaRoy
PERIOD COVERED: August 1-31, 1981
DATE OF REPORT: September 10, 1981

RELAXATION OF FILLER (L. R. Trentham and B. C. LaRoy)

Additional stress relaxation measurements of filler with normal and WS flavors are in progress. Initial observations confirm our earlier conclusion that WS treated filler has a higher short-term modulus than the normal material. However, sample-to-sample variations are large and more data are needed to quantitate the effect.

PROPYLENE GLYCOL¹ (J. C. Crump)

In the interest of understanding observed losses in weight of tobacco samples sprayed with propylene glycol, samples of tobacco sprayed with different concentrations of PG and pure PG samples are being monitored under different conditions of relative humidity. Initial results indicate that after thirty days, PG placed in the laboratory atmosphere or in an Aminco chamber has continued to lose weight. The chamber and room are at equilibrium and the PG vaporizes. However, when placed in a sealed chamber with an NaBr salt solution used to maintain a 58% relative humidity, the PG has gained weight during the thirty days. The PG is evidently picking up excess moisture from the system, water being more mobile than PG, and it is expected that a moisture equilibrium will eventually be reached after which the transfer of water molecules will cease and the NaBr solution will likely sorb PG, causing a weight loss in the PG solution.

VIBRATION TESTING OF CIGARETTES² (D. A. Full)

Tests of monitor cigarettes at a frequency of 100 Hz and a test duration of 17 min. were reported last month. This work has been extended to frequencies of 50 Hz and 200 Hz, accelerations from 16G to 91G, and to times from 15 sec. to ~3 hrs, with the following results:

1. At a fixed frequency and amplitude (fixed acceleration) weight loss, ΔW , as a function of time, t , can be represented by

$$\Delta W = At^B$$

where A and B are constants.

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2. If frequency and amplitude are varied between tests in such a way as to produce the same acceleration, then the parameter B above decreases with increasing frequency (decreasing amplitude).

The diminished sensitivity to duration at the higher frequencies was unexpected. It may be related to the fact that the wrap paper is not perfectly cylindrical, but has numerous local depressions with dimensions which are comparable to the amplitude of vibration. The disintegration of the filler rod is often observed to involve sliding as a monolith, within the wrap paper, in an incremental fashion. The size of these increments must be comparable to the amplitude of vibration, which is smaller at higher frequency, for a specified acceleration. If these do not exceed the characteristic dimensions of the depressions in the wrap paper, the likelihood is greater that successive incremental sliding motions will be simply back and forth rather than a "random walk."

WAREHOUSE CONDITIONS (B. C. LaRoy, J. C. Crump, M. J. Wood)

Monitoring of temperature and humidity in Cabarrus County and Richmond warehouses has continued. Malfunctions in instrumentation used to read the strip chart records has necessitated reanalysis of some of the data. An initial report is in preparation. Eight additional temperature/humidity recorders were received and calibrated.

On August 28, 1981, four hogsheads of bright (two grades) and two of burley (one grade) were opened and inspected in Cabarrus warehouse #6. No visible mold was seen and the tobacco seems in good condition.

RL-MOLD STUDY^{3,4} (J. C. Crump)

Samples of RL with various cooked flavor, PG and sorbate contents as supplied by L. Wu have been stored at 75°F and humidities ranging from 58% to 80% RH. The 75°F moisture isotherm was determined for each sample and the samples are checked regularly for mold. After 70 days, no macroscopic mold has been observed on any of the samples.

COMPUTER MODELING APPLICATIONS (H. A. Hartung, M. J. Wood)

Programs were written to interface APL modeling applications with new graphics capabilities on the DEC computer. We are now attempting to organize a special interest group (SIG) to coordinate the checking, debugging, documenting and training that necessarily goes with new computer applications.

At Westab we have been working on two applications of the IBM 5120 computer. One is the mass balance program and it was completed in August. All data on production, by-products, waste and inventory changes are stored in files on diskettes. The data are used to provide weekly and monthly summaries of yields and accountability. The second project at Westab is updating the system that tracks Q.A. and process data. This is scheduled for implementation in October. Records of about 60 parameters will be retained on diskettes on a shift-by-shift basis. Ten separate daily, weekly,

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and quarterly reports will be generated from these files. Historical files will also be kept on the 5120 and Westab will be freed from its current dependence on the old Sigma computer at R&D. The diskette files will be formatted so that they can be transmitted to the DEC computer for math modeling work.


SEMINARS (B. C. LaRoy and D. A. Full)

A series of three internal seminars was presented on the mechanical properties of viscous materials and tobacco. These were well received and provided the bases for numerous inter-project and divisional discussions.

REFERENCES

1. J. C. Crump, Notebook #7220, p. 139
2. D. A. Full, Notebook #7199
3. J. C. Crump, Notebook #7220, p. 139
4. J. C. Crump, RL-Mold Study, memo to L. Wu, Sept. 9, 1981
5. H. A. Hartung, et al, APL/Graphics "SIG", memo to distribution, August 24, 1981

/hws



2022151436

CHARGE NUMBER: 1716
PROJECT TITLE: Tobacco Microstructure
PROJECT LEADER: Mary Ellen Counts
PERIOD COVERED: August 1-31, 1981
DATE OF REPORT: September 9, 1981

Ca⁴⁵ AUTORADIOGRAPHY (S. E. Taylor, M. E. Counts)

Growing of bright 319 hydroponically in a nutrient solution to which the radioisotope calcium-45 was added has been successful. Bottom leaves are now being harvested. Several whole-leaf film autoradiographs have been obtained for these leaves. There appeared to be little difference in Ca⁴⁵ isotope distribution in the bottom leaves of plants fed all the Ca⁴⁵ dose at the beginning of the plant's growth and leaves from plants fed the isotope at increments during the plant's growth. The activity was primarily at the periphery of the leaves with relatively little activity in veins and mid-rib. Sections of leaf tissue were taken from several leaves and counted for activity in a scintillation counter. Although these measurements are qualitative, relative activity measured with the scintillator corresponded well with the calcium isotope distribution indicated by the autoradiographs.

These autoradiographs were obtained for uncured leaves. Similar autoradiographs and scintillation countings will be taken for leaves after curing. These tests will also be done with leaves of the upper stalk positions.

A tissue freezing liquid nitrogen refrigerator was obtained for long-term storage of the labelled tissue. Thin sections of tissue will be prepared for isotope location with emulsion autoradiography.

RATOONED TOBACCO (M. E. Counts, S. E. Taylor)

Field-grown control 319 bright tobacco and ratooned 319 bright tobacco from the 1980 crop year which had been cured and expanded were examined for surface morphology. The results were compared to a similar study for the 1979 crop year, and the data were in general agreement. There was a considerable variation in upper epiderm cell size and shape for filler from a particular stalk position. The average cell area for the lower stalk positions of the ratooned and control tobacco filler was generally greater than that for filler from the upper stalk positions. It was interesting that these averages of cell size were not very different for filler from top to lower mid-stalk positions. This was similar to the trend seen for the 1979 field-grown crop. The greenhouse control and ratooned leaves, however, showed a more defined increase in average cell size area from top to

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bottom stalk positions. These differences in field-grown and greenhouse-grown tobacco cell size measurements will be investigated further.

Mary Ellen Counts

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2022151438

CHARGE NUMBER : 1801

PROJECT TITLE : Tobacco Processing

PERIOD COVERED: August 1-31, 1981

PROJECT LEADER: F. V. Utsch

I. REORDERING OF EXPANDED PRODUCTS

A. Reordering Cylinder Optimization (D. R. Fox)

The three-week MC reordering test grid has been completed. This test is the culmination of considerable effort in developing an understanding of the effects of reordering time, cylinder throughput, and cylinder loading on product CV and shred size.

Analysis and modelling of the test data are currently in progress. However, initial results are very encouraging in that the models appear to agree with the tentative conclusions reached from previous testing. The large decrease in product CV with increasing cylinder throughput was again observed. An increase in residence time in the cylinder improved CV's if the cylinder loading was held constant by reducing throughput. If throughput was held constant, the increase in residence time resulted in a reduction of product CV. These results indicate that residence time and cylinder loading are the two major variables affecting product filling power. Of the two, the loading effect is more significant than the residence time effect.

A report will be prepared after data analysis is completed. Preliminary conclusions are being transmitted to MC and Westab personnel.

B. Improved Reordering Methods (C. M. Wicks)

The technique of using humid air to add the final moisture to partially reordered DIET was investigated. DIET material was spray reordered to 5, 7, 9 and 11% OV and then exposed to humid air to bring the moisture to 11%. The data contains quite a bit of scatter; however, a general conclusion is that the average CCV loss using humid air after spraying is 4 CV units. The data scatter was too large to allow conclusions concerning the CV loss at the individual OV levels.

II. DIET CLUMP SEPARATION (R. Z. Burde, P. E. Aument)

A series of four replicate tests comparing the effectiveness of the experimental clumpbreaking units for separating large (100 pound) DIET clumps was completed.¹ The results showed that the squirrel cage unit and the inclined pinned conveyor were the most promising candidates, with only about a 3% reduction in the longs + mediums combined sieve fractions. Product breakage on the Rotex unit was substantial and the unit has been returned to the supplier. Evaluation of the inclined pinned conveyor in line with the Phase III impregnator is planned beginning in early October.

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III. CO₂ IMPREGNATION STUDIES (B. Donenfeld)

A. Presnowing and Gaseous Impregnation

Outside consultants in the cryogenic fields were consulted about the properties of dry ice snow. The CO₂ snow manufactured by the 4 cone snow head and fire extinguisher nozzles is coarse and agglomerates rapidly upon contact. Current approaches to obtaining a free-falling uniform snow include "granulation" of dry ice blocks and using CO₂ mists as produced in freezer tunnels.

B. Breakage During Impregnation

A vibrating conveyor in D Pilot Plant was fitted with an 8-mesh screen. A quantity of DBC bright blend was run over the screen to obtain a feedstock of 100% longs at 20% OV. Replicate batches of ambient degassed and insulated degassed liquid impregnated tobacco and thawed, freezer frozen tobacco are being submitted for sieves.

IV. EXPANSION TOWER DESIGN STUDIES (B. Donenfeld)

A. Particle Temperature Determination²

The test of the Hughes IR instrument on the 8" rectangular tower has been scheduled for September 9 & 10.

B. 3" Tower

A design request was submitted to Engineering Services to equip the 3-inch tower with an 800°F running capability. Replacement of the blower, cyclone separator and heater are planned.

V. FREEZE DRYING OF TOBACCO (B. Donenfeld)

An attempt was made to determine the freezing point of 20% OV DBC bright tobacco. The sample, contained in an insulated vessel, was frozen in a -100°F chest freezer. Three thermocouples provided an average tobacco bed temperature. A weight and LVDT cell were placed on the bed and a two-pen recorder traced the bed temperature and LVDT cell output. It had been hoped that as the tobacco thawed, the bed would become pliable and show an inflection in the LVDT output. Instead, a gradual settling took place as the temperature rose from -100° to +32°F. When removed, the tobacco was obviously thawed. Other avenues are being investigated for determining the freezing point range.

VI. WS REMOVAL (B. Donenfeld)³

Thermal treatment was evaluated as a means of removing WS-14 from filler. Both dry heat and steam were used. Each method was successful in removing over 90% of the WS. The dry heat required over 30 minutes at 300°F. Steam stripping was much more effective time-wise and the results indicated that steam distillation would be effective in WS recovery.

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VII. DIELECTRIC DRYING OF RCB SHEET (R. Z. Burde, P. E. Aument)

Two rental units were installed in line-in the BL Pilot Plant. Tests are in progress and some 600 samples have been collected to date.⁴ The product is being evaluated for the degree of drying and its dependence on the initial input moisture, the effect of drying on the chemical constituents and moisture distribution throughout the sheet, and the physical characteristics of the sheet (CV, surface cracks, etc.).

Observation of the tests in progress lead to the following tentative conclusions, which need to be confirmed by complete evaluation of data:

1. The 12 1/2 KW units can only deliver up to 4 KW into the product. Thus the limiting factor is the ability of the product to accept the rapid surge of energy. This indicates that several low power units coupled together would be superior to one high power unit.
2. On the units tested, the maximum moisture reduction was from 30% to about 16% OV. Based on these findings, an additional unit is being rented to allow for a more complete drying study.
3. The effectiveness of the units is related to the initial RCB sheet moisture. This confirms the initial findings that dielectric treatment can improve moisture distribution in the sheet.
4. Excessive power input leads to arcing. Addition of a third unit and placement of a "hold-down" conveyor should greatly improve the situation.

VIII. OTHER EFFORTS

The large Triple S Dynamics air table testing showed only a 50% efficiency for removing buggy whips and an 83.4% product recovery at 250 lbs/hr. The testing was therefore discontinued. The salt ball removal effort also showed little promise. A summary and background of the pilot scale WET process development efforts was prepared and issued.^{5,6}

IX. REFERENCES AND MEMO'S ISSUED

1. Memo to Mr. F. V. Utsch from P. E. Aument and R. Z. de la Burde "Evaluation of Clumpbreaking Methods", August 19, 1981.
2. Memo to K. S. Burns from B. Donenfeld, "Particle Temperature Determination", August 26, 1981.
3. Memo to K. S. Burns from B. Donenfeld, "Thermal Treatment for Removal of WS", August 27, 1981.
4. Development Notebook 7685 pages 1-65, P. E. Aument.

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5. Memo to K. S. Burns from P. E. Aument and R. Z. de la Burde, "Wet Process - Historical Perspective", June 29, 1981.
6. Letter to Mr. Robert M. Shaw of Fish and Neave regarding WET background and experimental results, from F. V. Utsch dated July 29, 1981.
7. Special Report, "Reduction of Residual Ammonia from Westab ET Using an Air Sweep", Accession No. 81-191 by P. E. Aument, August 13, 1981.

F.V. Utsch

/deb

2022151442

CHARGE NUMBER : 1804

PROGRAM TITLE : Expanded Tobacco - Process Improvement

PERIOD COVERED: August 1-31, 1981

PROJECT LEADER: R. G. Uhl

I. TOWER STUDIES (H. X. Nguyen)

Initial trials were conducted to evaluate the 4" x 14" rectangular expansion tower which replaced the 8" diameter round tower in the Phase III system. The rectangular cross-section was installed to reduce tobacco roping in the elbows, thereby improving heat transfer and product uniformity.

Runs made at the standard pilot process gas temperature of 600°F gave disappointing results, with tower exit OV's at 4-5% and equilibrated CV's at 55-60 cc/10 g. Tower temperatures were raised in increments up to a maximum of 740°F, with results showing that significantly higher temperatures (+100°F) were required to achieve CV parity with round tower product. However, the rectangular tower product results lie along the same CV versus tower exit OV and equilibrated CV versus equilibrated OV curves as round tower product, indicating that the higher temperature is necessary to obtain the same degree of tobacco thermal treatment.

Raising gas velocity from 75 to 175 fps caused a 10 unit product CV decrease, as opposed to a 10 unit increase for the same change made in the round tower. The rectangular tower had been fitted with a pronounced (75% area reduction) venturi feed section which caused large negative pressures at the feed throat and in the tangential separator, particularly at higher velocities. Reducing the tobacco feed rate had little effect on CV, but doubling the normal feed rate caused a 10-15 unit CV loss.

The venturi was replaced by a conventional feed section which reestablished the normal tower pressure profile. Higher gas temperatures were still required to achieve targeted CV levels. Velocity variation between 75 and 225 fps gave little change in tower exit OV or CV. Sieve fractions of the rectangular tower product show a long fraction 10 units higher than round tower product at the same tower exit OV. The standard 16" radius elbows have been replaced by the short (8") radius elbows with special I-R windows to attempt infrared measurement of tobacco particle temperatures.

Evaluation of the retractable arm sprayer was completed. Action of the arms was slowed by the low temperatures. Seal leakage caused partial retraction of the arms during impregnation, and the arms themselves lost strength and bent down under the tobacco load after a few batches. High actuation pressures were required to retract the arms from the tobacco bed. Nevertheless, although a total batch cycle could not be evaluated, visual observation indicated that this approach could reduce batch compaction if it could be made mechanically reliable.

Two of the three MC clumpbreakers have had their rotational speed reduced to 20 rpm. Samples are being taken for sieve analysis.

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II. HEAT TREATMENT (A. L. Johnson)

Experiments investigating tartaric acid and ammonium tartrate pretreatment of tobacco to reduce darkening were completed. There was no discernable difference in the color of pretreated and non-pretreated products. Product CV's were also similar (Eq. CV/OV = 45/9.5).

Thin and bodied portions of the DBC bright blend were treated separately to discern any differences in color and CV. For any given treatment time the bodied product was darker than the DBC blend, while the thin portion was essentially equivalent in color to the blend. The thin and bodied portions were as susceptible to "salt and pepper" coloring as the DBC blend itself. The thin portion of the blend lost its inherent 5 CV unit advantage during treatment, with both products having identical Eq. CV/OV values of 51/9.0.

III. PERKS (A. L. Johnson)

Efforts to scale-up the Project 1503 PERKS process continued. Additional preliminary trials showed that the processing of RKS at the desired 75% OV content is not feasible with the existing conveyor/dryer system due to losses from sticking to belts, etc. Tests showed that 55-60% would be a workable add-on level in this respect and that more concentrated lime slurries could be sprayed successfully. Pump and nozzle components of materials compatible with peroxide were received and installed.

A first effort at making a complete PERKS run was made using a more concentrated chemical spray (attained by reducing water addition to the slurry) in order to achieve the desired chemical add-on levels at an OV of 60%, both to facilitate handling of the RKS on the equipment and to reduce the sizable drying load (75% OV material would reduce dryer capacity to 10% of normal roaster throughput). Reactant mixture bubbling and ammonia evolution were much more pronounced in the concentrated mixture, and probably spent the peroxide. Project 1503 will attempt to provide a chemical addition system within the 60% OV constraint.

A. L. Johnson

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2022151444

CHARGE NUMBER: 1901
PROJECT TITLE: Biochemical Modification of Tobacco
PERIOD COVERED: August 1-31, 1981
WRITTEN BY: D. M. Teng
DATE OF REPORT: September 9, 1981

I. LANCASTER LEAF TOBACCO COMPANY (LLTC)-TYPE FERMENTATION OF BURLEY TOBACCO (D. Teng, S. Tenhet)¹

Small scale solid fermentation of Burley strips and stems were carried out at R&D to determine the effect of sugar concentrations to the degree of nitrate removal from the materials. Samples were treated with water or different concentrations of sugar, and kept at 50°C for up to three weeks. Samples were dried, ground and submitted for analysis. Results will be reported upon the completion of the analysis.

II. FARM STUDY

Coker 319 Bright tobacco leaves from mid-stalk position were primed and flue cured at the Southern Piedmont Experiment Station at Blackstone, Virginia. Some plants were also stalked out and are being air cured. These samples will be used in a model study to determine the effect of reducing sugar to filling power and expandability. (D. Teng, G. Nixon, K. Walls)²

Samples were taken at different stages of flue curing and air curing, and the changes in pectinmethylesterase activity were determined. On dry tobacco weight basis, the enzyme profile from the mid-stalk leaves was very similar to that of the bottom-stalk leaves. The enzyme activity change could be attributed to temperature, time and moisture content of the leaves being cured. (G. Nixon, K. Walls, E. Mooz)²

A study of Burley tobacco was initiated at a farm in Abingdon, Virginia. Burley plants were stalk cut and subjected to air curing. Leaves were also primed and shipped to Southern Piedmont Station to be flue cured. This set of samples also will be used in a model study to determine the effect of reducing sugar on filling power and expandability. Chemical analysis, smoke chemistry and subjective characteristics also will be determined on all samples. (D. Teng)

III. TOBACCO EXPANSION

Burley strips were heated by a small scale radiant heat system, equilibrated and shredded. The equilibrated C.V.'s with/without the

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radiant heat treatment were determined. No significant difference was observed. On the other hand, Burley cut filler which was treated by the radiant heat showed a significant increase in filling power as compared with the non-radiant heat treated filler. (E. Mooz, K. Walls)³

Tobacco fillers (total blend) from the Manufacturing Center Primary were treated by the radiant heat system. The samples taken before and after the ADT dryer showed very significant increases in filling power by the treatment. The experiments will be repeated to verify the results. (E. Mooz, K. Walls, D. Teng)³

REFERENCES

1. S. Tenhet - PM Notebook #7225.
2. K. Walls/G. Nixon - PM Notebook #7528.
3. E. Mooz - PM Notebook #7443.

/11v

D. Teng

2022151446

CHARGE NUMBER: 1902
PROJECT TITLE: Microbial Technology
PERIOD COVERED: September 1-30, 1981
WRITTEN BY: V. S. Malik *VS Malik*
DATE OF REPORT: September 2, 1981

I. GROWTH STUDY WITH ATCC 1 (D. Chadick)

The growth of ATCC 1 was studied in nitrate broth, TSB with KNO_3 (1% w/v), and pasteurized SEL. Growth was similar in nitrate broth and TSB. However, there was a slight decrease in growth in the SEL. These results were supported by the number of viable cells obtained in each medium as judged by plating on solidified agar.

II. ISOLATION OF THERMOPHILIC DENITRIFIERS (D. Chadick)

Biochemical testing has continued with all isolates. A nine-media battery was set up and incubated at 50C for 24 hrs. There were basically no differences in PM 5-10 and ATCC 1, 3, and 4 based on these results. Further testing is required in order to find media that may differentiate between isolates that otherwise appear alike.

III. PLATING OF TANK H (D. Chadick)

Plating of tank H is continuing. Viable counts continue to be in the range of 10^6 to 10^7 bacteria per ml with the predominant organisms being phenotypically similar to ATCC 1.

IV. THERMOPHILIC DENITRIFIERS ISOLATED AT ATCC (D. Naugle)

Agar slant cultures of 19 isolates were received from ATCC. Agar slants and stabs were made of each isolate. The isolates were also plated to check for purity and to determine phenotypical appearances. All isolates were tested for their ability to denitrify. Results for the ability to denitrify corresponded with those received from ATCC. However, there was one isolate which was found to denitrify in two separate tests but was reported by ATCC as a non-denitrifier. Of all isolates received from ATCC, 22 have been found to denitrify; 5 isolates are non-denitrifiers. All grow at 50°C.

V. STORAGE OF CULTURES (D. Naugle)

The liquid nitrogen storage system is in operation. All isolates received from ATCC have been frozen after adding 5% dimethylsulfoxide. A regular system for nitrogen tank delivery has been arranged.

ATCC = American Type Culture Collection.

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V. PILOT PLANT (H. Bravo)

At pilot plant "C", tank H was operated in the semi-continuous mode. Batches of denitrified SEL were produced to make reconstituted tobacco for chemical, biological and panel tests. In this tank sedimentation experiments showed a uniform cell density distribution throughout the tank when 157 and 350 gallons of inoculum were used. However, a sedimentation gradient was found only when 157 gallons of inoculum was used.

Under varying parameters, the 500 gallon tank was found to denitrify 50 gal/hour of 4000 ppm SEL.

In a 500-liter fermenter inoculum start-up studies using the plauxostat set-up indicate the feasibility of the approach, however, more laboratory studies are required.

Studies on preservation of large amounts of these inoculum indicate that inoculum stored at room temperature and with KNO_3 has no foul odor and is active after six days storage.

The carbon source in SEL was found to be mostly in the soluble form.

A preliminary production plant cost estimate is in progress.

VI. STARTING UP DENITRIFICATION (P. Oglesby)

The start-up process was implemented into the 500-liter fermenter using an initial pH of 7.2. This proved successful except that it was noted that the pH dropped initially. Once the cell count increases, the pH rises and the process starts. This usually occurs after 16-20 hours. Start-up experiment tests were conducted in 14-liter fermenters to determine the optimum pH for start-up. Tests were done at 6.5, 7.0 and 7.5 using 1% inoculum. It was demonstrated that pH 7.0 is the best of the three. pH 6.5 started first during the test but did not consistently denitrify the SEL as quickly as pH 7.0. pH 7.0 achieves full-scale denitrification at a much faster rate than the other pHs and maintains this throughout the run. Further testing will be conducted to optimize the start-up procedure.

VII. GROWTH MEDIUM FOR PURE ISOLATES (S. Grable)

Isolates PM5-29 are being grown in Luria broth at 55°C with moderate shaking. In the same manner ATCC 1 is being grown for comparison. All are monitored hourly for optical density and nitrate disappearance. Thus far, several of the isolates appear to be similar if not the same.

Several media have been compared for the growth of the various bacteria. Luria broth appears to support the best growth.

/hws

2022151448

CHARGE NUMBER: 1990
PROJECT TITLE: Blend Development
PROJECT LEADER: C. Moogalian
PERIOD COVERED: August, 1981

Blend development for new products and any necessary brand modifications will be the primary objectives for this newly-formed group. To help meet these objectives, basic studies with tobaccos and processing techniques will be correlated with their influence on various taste factors. The Blend Development Group will function in close association with the Leaf Department, and will assist other groups working with blend-related projects.

In addition to the above, blend development for Project Tomorrow and the Burley Reduction Program will be continued within this group.

I. PROJECT TOMORROW

The objective of this project is to develop a prototype(s) that will achieve a 60:40 preference among Marlboro smokers when tested against Marlboro.

Final preference results on the first three models to be POL tested in this project have been received. Results from those smokers expressing a preference are listed below. Final reports will be available in September.

	Percentages	
	<u>Marlboro</u>	<u>3157</u>
85 mm Marlboro smokers	50.6	49.4
Other 85 mm FF smokers	50.6	49.4
	<u>Marlboro</u>	<u>3158</u>
85 mm Marlboro smokers	58.9	41.1
Other 85 mm FF smokers	52.2	47.8
	<u>Marlboro</u>	<u>3160</u>
85 mm Marlboro smokers	44.4	55.6
Other 85 mm FF smokers	54.3	45.7

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3157 - Contains expanded Oriental instead of expanded Bright; also, some regular Oriental.

3158 - One half of the blend is partially expanded Marlboro strip blend. No by-products.

3160 - Similar to 3157, but larger amount of expanded Oriental. No regular Oriental.

All the above models contain the Marlboro flavor system. We will submit 3160 without aftercut to the Flavor Development Division to obtain a more tailored aftercut. Some minor blend adjustments will also be investigated.

We have a modified version of 3158 which screened well against Marlboro. Harshness appears to be the objection to 3158 among the POL panelists. The modified version is considerably milder. It differs blendwise from 3158 in that it has half the level of partially expanded Marlboro strip blend and contains by-products.

Internal screenings of blends containing 15% of an expanded Bright-Oriental mixture have looked very promising. We will request POL's on two of these blends.

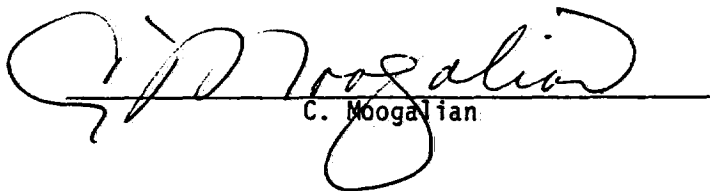
II. BURLEY REDUCTION PROGRAM

Due to a shortage of burley production the last two years and an expected shortage again this year, a program to help reduce burley usage in our products was initiated.

Marlboro, Benson & Hedges 100's, and Merit samples with 3% reduction in Burley (replaced by Bright) and 10% reduction in Burley spray rate were requested for POL tests after successful internal screenings. The Marlboro (3174) and the Benson & Hedges 100's (6035) have been made and shipped. The Merit (4235) is awaiting smokers. Preliminary results for the Marlboro and B&H 100's are expected in September.

III. MISCELLANEOUS

Two of our members, L. F. Filosa and C. Moogalian, participated with L. C. Jennings in a three-day seminar presented in the Leaf Department. Cigarette construction, flavor, and blend were topics of the seminar.


C. Moogalian

CM/1ad

2022151450

CHARGE NUMBER: 2100
PROJECT TITLE: New Products
PROJECT LEADER: W. G. Houck, Jr.
PERIOD COVERED: August, 1981

I. NEW PRODUCTS

A. MUL 100

Production start-up of both regular and menthol versions of the Merit Ultra Lights 100 mm began on August 17, 1981. Both cigarettes have been running well and meeting target specifications. Market distribution is scheduled for early October, 1981.

B. Project CAM II/III

Preliminary specifications for Project CAM II 85 mm (1 mg Cambridge with grooved filter) have been readied for possible test market start-up. Modeling for the 100 mm version is in progress with consumer testing to follow.

Particle fall-out continues to be a concern with the $Mg(OH)_2$ flavor carrier used in the CAM III phase of this project. In light of this, more emphasis will be placed on improving the special flavor release carbon from Pittsburg. Detectable flavor release has now been demonstrated after two cycles of accelerated aging with both flavor carriers.

C. Project GROW

VP testing¹ of a 0.5 mg Project GROW candidate vs. Barclay was completed. However, due to low smoker group returns and inconsistencies in the data, this test is being rescheduled with a larger panel. The VP test² of the 0.5 mg Project GROW vs. the MF-5 candidate is ready for shipment.

II. INSTRUMENTATION

A. Tipping Permeability Instrument (TIPPER)

This instrument involves the combining and updating of the PDI instrument and the tipping paper pressure drop clamp. With the new combined instrument, designed specifically for tipping, both sides of the perforated tipping can be measured simultaneously. A prototype model has been fabricated with additional modification in progress.

III. REFERENCES

¹VP 4789

²VP 4795


W. G. Houck, Jr.

WGH/lad

2022151451

CHARGE NUMBER: 2105
PROJECT TITLE: Filter Development
PROJECT LEADER: W. A. Nichols
PERIOD COVERED: August, 1981

I. CAM II

Brown and Williamson's patent application for grooved filters discusses the necessity for an air or smoke impermeable plugwrap. To determine the limits of permeability to smoke nonporous plugwrap, Ecusta 648, was tested. Using a 1.13 cm² exposed paper area, smoke was drawn through the plugwrap by a syringe pump (1050 cc/min.) Twenty-five samples were tested and visible smoke flow could be seen with all samples.

II. EXTRUDED TOBACCO

Work continued on the evaluation of various binders. Sheet samples were produced with various molecular weight Klucels and polyvinyl alcohol. Satisfactory sheet was made with 50% solids (4% Klucel, 4% polyvinyl alcohol, 92% tobacco dust) and 50% water. This formulation represents the most economical sheet yet produced. Further sheet properties will be evaluated.

III. CIRCUMFERENCE MEASUREMENT

Testing of a new design that replaced the Eastman LaserMike[®] modification was completed. A unit will be made available to QA for factory testing.

IV. ADHESIVE APPLICATION

A Findley adhesive foamer was installed and tested. In collaboration with Manufacturing Operations, foamed tipping adhesives are being evaluated as a method to reduce adhesive usage and possibly eliminate skip tip adhesive application. Tests were performed with 50% foamed adhesive on a PA-8 at 4200 cigarettes/min. Satisfactory adhesion was achieved with skip tip and full coverage adhesive application.



W. A. Nichols

WAN/lad

2022151452

PROJECT CHARGE: 2106

PROJECT TITLE: APPLIED TECHNOLOGY

PROJECT LEADER: Peter Martin

WRITTEN BY: Peter Martin and J. Banyasz

PERIOD COVERED: August 1 - 31, 1981

DATE OF REPORT: September 10, 1981

PROJECT GROW

Together with W. Mutter's Division and outside consultants the Puff Profile Analyzer has been very carefully examined to verify its operation. No major problems were found and the unit has been used in a demonstration of the Barclay dilution mechanism to outside consultants.

NATURALLY OCCURRING DENITRIFICATION

The series of Fed-batch runs in the pilot plant with the mixed culture inoculum derived from a phauxostat have been completed. They are currently being analyzed to show the effect of different SEL feed rates and differing seed levels of inoculum. Extremely good reproducibility was obtained with pasturized SEL and accurately controlled temperature.

A presentation on the results so far was made to Helmut Gaisch and Deiter Schultes of P.M. Europe. Several meetings were held with them to evaluate additional information needed to make this a viable process.

A project was undertaken in collaboration with B. Semp (Mfg.) to characterize the organism primarily responsible for denitration, to define the optimal inoculum and to obtain the data necessary to design an inoculum feed line.

The results of the biochemical characterization tests furnished by ATCC on the submitted isolates were examined. A comparison showed that ATCC-1 was 85% biochemically similar to a later isolate from the Fed-batch reactor identified by ATCC as Bacillus circulans. The matches between ATCC-1 and other identified isolates were weak. A culture of Bacillus circulans was grown up and was shown to reduce NO_3^- to N_2 in a phauxostat.

Collaborative work with B. Semp was also begun on a synthetic start up medium. V. Malek had shown that ATCC-1 can be successfully started in a 10% solution of SEL in water. To avoid the problems inherent in the storage of large volumes of SEL some SEL was concentrated to 70% dissolved solids. This concentrate minimized both volume and spoilage problems. A medium containing glucose and NH_4NO_3 was prepared. Shaker flasks containing this medium as well as tobacco solubles ranging in concentration from 0.05 to 1% as added from the

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concentrate were inoculated with ATCC-1. Growth was observed in all flasks and appeared to be proportional to the concentration of tobacco solubles over the range tested.

HEAT TREATMENT OF TOBACCO MATERIALS

Work has continued on developing the phase diagram of bright, burley and oriental tobacco. A problem with Karl Fisher determinations of the expanded samples has been solved.

Oriental tobacco has been heated at low temperatures (135-200°F) for varying lengths of time with no apparent pattern for CV increases. Work is continuing at higher temperatures.

ADDITIONAL PROJECTS

It was found that there was insufficient contrast and regularity of the dark bands on the BL sheet to allow accurate quantifications of their intensity and frequency using the rotating wheel and the Fast Fourier Transform oscilloscope. A system is being set up to scan any future sample for the spatial distribution of density.

Peter Martin
J. L. Baryas

/ev

2022151454

CHARGE NUMBER: 2305
PROJECT TITLE: Flavor Development
PROJECT LEADER: C. N. Kounnas
PERIOD COVERED: August, 1981
WRITTEN BY: J. L. Spruill
DATE OF REPORT: September 8, 1981

I. Low Delivery Program

Production samples of 100 mm Merit Ultra Lights regular and menthol are being monitored subjectively.

Preliminary results from POL 7073 appear good for a 100 mm modified MUL candidate vs. Winston Ultra in the MF-5 program.

Recommended cigarette models for Project Grow are pending fabrication. Final POL reports are being written for 100 mm Cambridge Menthol testing.

Three POL tests were requested for the Low Lamina Blend program, using RL-11, RL-13 and RL-150B vs. Merit.

II. After-cut Flavor and Casing Substitution

Several POL tests involving casing and after-cut chocolate and MS flavor are pending cigarettes. POL tests have been requested for vanilla replacement.

III. Distinctively-Flavored Cigarettes

Twelve HTI tests of Northwind vs. P.M. and competitive brands were requested by Marketing, along with a series of mini-mall studies.

Most of the Summit testing is being closed out and reports written. POL test 9035 (9 mg. Merit-type 100 WS vs. Salem Lights 100) will be shipped in September.

Field pick-ups of Northwind test market samples are subjectively acceptable. WS monitoring in the plant shows no migration. Work is continuing on emulsification of WS in A/C. WS material specifications have been revised.

IV. Special Investigations

A report is being written on the WS accelerated aging study. Work in the menthol program continues pending remake of some samples.

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V. Services to Other Groups

Flavors made by MC for domestic and overseas cigarette manufacturing are under evaluation.

Subjective evaluations on some PM brands using Det PG have been completed.

VI. References

Notebook 7565, pp. 128-36
Notebook 7569, pp. 121-28
Notebook 7608, pp. 138-150
Notebook 7522, pp. 55-58
Notebook 7663, pp. 44-84
Notebook 7681, pp. 1 - 8
Notebook 7599, pp. 107-117

J. L. Spruill

2022151456

CHARGE NUMBER: 2305
PROGRAM TITLE: Flavor Development
PROJECT LEADER: J. W. Swain
PERIOD COVERED: August 3 - 31, 1981
DATE OF REPORT: September 2, 1981
WRITTEN BY: J. W. Swain and L. S. Wu

I. RL Flavor

Internal evaluation of Project Read modifications of RL, RCB, and Burley casing in Marlboro indicate that additional flavor development is necessary prior to Consumer Panel Testing. Preliminary data on V-3848 testing the Project Read modification of Burley spray in the Y79-3 Marlboro show promising results. Additional preservatives are currently being evaluated in the modified RL and RCB sheets in the storage stability study.

II. Cooked Flavors

A factory trial of the Y83-1 Marlboro test market blend was made at Stockton Street on August 25 and 26. The RL-C (Cooked Flavor) was produced at Park 500 on August 13 and 14.

Part of the test blend, as well as a control blend (Y79-3) were retained for filling power studies at R&D. Another part of the blend was made into cigarettes at Stockton Street at three different weights. The cigarettes and blends are under evaluation.

Preliminary results of POL 3168, testing the Y79-3 Marlboro control vs. a Y83-1 minimum change Marlboro test showed no significant differences. The status of the other consumer panel tests are listed below:

N-3169 - in the field
N-4219 - in the field
N-4220 - in the field

III. Services to Other Groups

Evaluations are in progress on water expanded tobacco (WET) generated at Westab. After comparing 100% cigarettes of four WET samples to ET and DET, the WET-1 and WET-4 were selected for evaluation at 25% levels in an Australian blend.

Flavor Group screening of N-3170 comparing 15% Carbamate ET to 15% ET and N-3171 comparing 15% Carbamate ET to 15% DIET showed significant differences in flavor and response.

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IV. References

1. Notebook #7564, pp. 159-164
2. Notebook #7614, pp. 38-43
3. Notebook #7657, pp. 71-80

J. D. Swain
Louise Wu

2022151458

CHARGE NUMBER: 2306
PROGRAM TITLE: Flavor Component Evaluation
PROJECT LEADER: R. M. Ikeda
PERIOD COVERED: July 1 - August 31, 1981
DATE OF REPORT: September 8, 1981
WRITTEN BY: R. M. Ikeda

I. Tobacco Flavor¹

U. S. Bright tobacco was sequentially extracted with methylene chloride and then with ethanol. The ethanol extract was injected into 100% RL cigarettes and was found to reduce the burnt woody character and a much smoother smoke. Ethanol was removed and the residue partitioned between water and methylene chloride. Only the methylene chloride phase contained the smoothing components. The methylene chloride fraction was separated by silicic acid column chromatography. Three fractions were eluted with hexane, methylene chloride and methanol. Only the methanol eluted fraction contained the material that improved the flavor of the RL cigarettes. Reverse phase HPLC analysis of this methanol fraction showed the presence of a large number of components.

Fraction 5 isolated from the volatile neutral material from U. S. Bright tobacco was found to improve the smoke flavor of low delivery Bright cigarettes. This fraction was separated by packed column gas chromatography and 14 of the odorous "peaks" were trapped. The trapped materials are being evaluated by members of the Flavor Development Division for their odor intensities and combination that can restore the odor of the original fraction. A 50 m capillary column was installed in the gas chromatograph and a splitter was attached to the end of the column to allow for odor evaluations of the separated components. Initial evaluations were run on fractions separated by column chromatography of the original volatile neutral fraction.

II. Flavors and Casings²

The Carbowax coated fused silica capillary columns deteriorated quite rapidly when used in conjunction with the Purge and Trap device for the analysis of the volatile components of tobacco and filler. Because of this problem, a bonded phase silicone capillary column was installed. One problem with the silicone column was the extinguishing of the flame just after the GC scan was started. The problem appears to be the large amount of water that condenses in the Tenax trap. The tobacco sample was purged for 10 minutes, an empty tube was substituted for the tobacco sample and purged an additional 30 minutes. This additional purge time should remove most of the water condensed in the Tenax trap. The GC analyses by this modified procedure eliminated the flame out problem. The modified procedure also eliminated some variable retention time peaks early in the GC scans.

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III. References

1. Notebook #7574, pp. 61-71
Notebook #7483, pp. 148-162
2. Notebook #7464, pp. 183-200
Notebook #7674, pp. 1-27

R. M. Ikeda

2022151460

CHARGE NUMBER: 2308
PROGRAM TITLE: Room Aroma Component Evaluation
PERIOD COVERED: August, 1981
PROJECT LEADER: A. G. Kallianos
DATE OF REPORT: September 10, 1981

I. SIDESTREAM SMOKE EVALUATIONS*

Pursuant to studies leading to a determination of the threshold level of WS-14 in sidestream smoke, we have obtained subjective descriptions of the relative characteristics of sidestream smoke from Northwind and Merit cigarettes. The assessments were made utilizing the room aroma evaluation apparatus. The panel, consisting of twenty people, was able to distinguish subjectively the two sidestreams of smoke over a wide range of concentrations. And, although the level of recognition of either cigarette increased as its relative concentration increased, the quality of odor did not appear to change very much, except that WS cooling becomes more obvious at higher concentrations.

The composite of sensations derived when sniffing sidestream smoke include odor, pungency, and irritation of the eyes and mucous membranes. In general, the panel found sidestream smoke from Northwind cigarettes to be less objectionable than that from Merit. Less smokey, less pungent, fresher, cooler, and even less irritating were the adjectives used to describe Northwind sidestream smoke relative to Merit. At high concentrations, where WS cooling is obviously apparent, a tingling, penetrating sensation was perceived in the throat with a cooling, stinging sensation in the eyes and sinus cavities. These were perceived as being "menthol" related and were not judged as objectionable. An overall change in the ammoniacal character of the smoke was noted providing a freshness reminiscent of "Windex" window cleaner.

The WS threshold measurements are nearing completion. Comparative studies with sidestream smoke from menthol cigarettes will follow.

II. COOKED FLAVORS

Work reported at this time relates to studies conducted by several groups within R & D in attempts to establish analytical specifications for cooked flavors and to develop procedures for quality control purposes. The complexity of the cooked flavor mixture and the limited knowledge regarding compositions and "active" flavor principles have prompted us to consider several procedures in this pursuit. A program has been outlined (memorandum by A. G. Kallianos to F. L. Daylor, August 3, 1981) to evaluate methods giving specific information on volatile and non-volatile components as well as methods which may lack specificity but could yield usable information.

Preliminary work has been conducted with nine samples. Six of these were considered acceptable on the basis of flavor evaluations and three were considered unacceptable, although some questions arose on the validity of these designations.

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Mr. H. Grubbs and his group examined the samples using HPLC in a reverse phase, without extraction or pre-analysis workup. Although tentative, their conclusion was that "fingerprint" chromatograms may provide information on the acceptability of a particular sample.

Ether extractable materials from these samples were prepared by D. Douglas and chromatographed on a fused silica glass capillary column coated with Carbowax 20M. His tentative conclusions, reported in a memorandum dated September 3, 1981 to R. Comes, state that a peak identified as isovaleric acid relates quantitatively to the acceptability of the samples, and that a similar correlation existed with a peak identified as valeric acid. Reported in that memorandum were also tentative identifications of methyl-, dimethyl-, and trimethylpyrazines. Responsibility for further work with volatile materials using high resolution gas chromatography was transferred to the Analytical Division.

We evaluated methylene chloride extractables from the nine samples, spiked with an internal standard, by gas chromatography using a series of packed columns. Although our chromatograms provided low resolution of materials, they showed delineations between acceptable and unacceptable samples by variations in the relative intensities of several peaks. Importantly though, we found that the intensities of many peaks could be influenced by the pH of the cooked flavor samples. With no apparent relationships between acceptable and unacceptable, the pH of the nine samples varied from 4.58 to 6.52. Adjustments of the pH of the samples to a common pH appeared to sharpen the delineation between acceptable and unacceptable samples on the basis of relative intensities of several peaks.

Aliquots of methylene chloride extractables from an acceptable sample were dissolved in ethanol and injected into cigarettes for flavor evaluation. Extractables were also obtained from aliquots of this sample after pH adjustments. In these instances, the pH was adjusted by a whole unit, both above and below the extant level. Aliquots of these extracts were injected into cigarettes for flavor evaluations. All three samples imparted desirable flavor smoking qualities to the cigarettes. Smoking evaluation of non-dialyzable pigment recovered from a cooked flavor preparation revealed undesirable flavor effects.

Early in the course of this work, we recognized the need for a more extensive array of samples, both acceptable and unacceptable, to evaluate variations, ranges, and the effects of different variables in the preparation of cooked flavors on the analytical outcome. For this purpose, Mrs. Louise Wu has prepared fourteen new samples; some expected to be acceptable and several intentionally "ruined." These samples are currently under investigation by various analytical procedures and subjective assessments. Specific gravity determinations and pH measurements have been completed on all of these samples. Neither specific gravity nor pH appear to correlate with acceptability, although pH measurements appear to provide a consistent reflection of the history of the samples. Sugar analysis and sensory evaluations have also been completed for seven of these samples.

Working with these seven samples, C. Kroustalis and F. Hsu have obtained ethyl acetate extracts which they have independently chromatographed using high resolution techniques. Based on GC retention times, Kroustalis has tentatively identified pyrazine, 2-methyl-pyrazine, 2,3-, 2,5-, and 2,6-dimethylpyrazines, 2,3,5-trimethylpyrazine, isovaleric acid, n-valeric acid, 2-ethyl-3,5- and 3,6-dimethylpyrazines and possibly acetylpyrazine. Additionally, he has found large variations

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on repeated scans within acceptable as well as intentionally ruined samples. However, there are indications of quantitative differences between acceptable and "ruined" samples. Interestingly, he observed that the ethyl acetate extracts of ruined samples were devoid of color. Hsu has obtained 35 profiles by replicate analyses of the seven samples using high resolution gas chromatography. Seventy-three peaks from each profile were selected as input to factor (BMDP4M) and discriminant (BMDP7M) analyses to separate pattern plots. Data analysis is in progress.

Gas chromatographic analysis of methylene chloride extracts from the seven samples on packed columns revealed rather complex chromatograms. These are being examined visually for significant distinguishing features.

Work on this aspect of the project will continue by the several groups.

III. REFERENCES

- *1. A. G. Kallianos, Notebook Number 7619, pp. 8, 9, 10.
2. B. T. Joyner, Notebook Number 7605.
3. L. Wu, Notebook Number 7639.
4. C. Kroustalis, Notebook Number 7274.
5. F. Hsu, Notebook Number 7576.



A. G. Kallianos

AGK:gmm

2022151463

CHARGE NUMBER: 2500
PROJECT TITLE: Synthesis of Tobacco Additives
PROJECT LEADER: J. I. Seeman
PERIOD COVERED: August 1-31, 1981
DATE OF REPORT: September 8, 1981
WRITTEN BY: C. G. Chavdarian

I. Nicotine Chemistry

For the first time, the individual enantiomers of 6-methylnicotine have been successfully prepared and purified.¹ Free radical methylation of (-)-nicotine with *t*-butylhydroperoxide and ferrous sulfate afforded (-)-6-methylnicotine. The same reaction with (+)-nicotine yielded (+)-6-methylnicotine. These compounds appear to be nearly optically pure. The specific rotation of (+)-6-methylnicotine was found to be +175°. These enantiomers are of great importance for our pharmacological and behavioral studies due to the fact that racemic 6-methylnicotine is known to possess pharmacological activity on a par with nicotine.

The major study concerning the methylation of nicotine is nearing completion. The product mixtures from eight of the methylation runs were subjected to HPLC purification. The components of each mixture were successfully separated and isolated, and optical rotations were obtained.² Methylolithium additions to nicotine result in partially racemized products with 6-methylnicotine undergoing the greatest racemization in most cases. On the other hand, radical methylation of nicotine provides products with high optical rotations. The radical methylation approach appears to be the method of choice for obtaining analogs in high enantiomeric excess.

A number of nicotinoids were prepared and purified for a variety of studies. Additional 6-(2-hydroxyethyl)nicotine was synthesized for the ongoing collaborative study into the use of affinity chromatography for nicotine receptor isolation.^{1,3} To assist us in an understanding of the mechanism of racemization during the addition of organolithium reagents to nicotine, 2'-deuteronicotine has been prepared.¹ A synthesis of 2'-methyl-nicotine free of the major byproduct, 6-methyl-2'-methylnicotine, was achieved.¹ The *cis*- and *trans*-5'-methylnornicotines were successfully separated and isolated by the use of HPLC.²

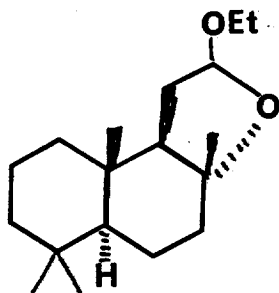
II. Flavor Chemistry

For odor evaluation by multidimensional scaling a total of fifteen 2-monoalkyl and 2,3-dialkyl pyridines and pyrazines were prepared.⁴

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Preparation of various 2,6-dialkylpyridines is also underway.⁵ By the use of alkylolithium reagents, 2-*t*-butyl-6-ethylpyridine and 2-*t*-butyl-6-isopropylpyridine were synthesized. Attempts are continuing in an effort to prepare 2-ethyl-6-methylpyridine and 2-isopropyl-6-methylpyridine.⁵

The woody odorant isolated from the photo-oxygenation of abienol has been tentatively identified as structure I through the use of NMR spectrometry carried out by R. Cox.⁴ Synthesis of the odorant is currently in progress. Reduction of sclariolide with DIBAL provided the lactol precursor to I as an epimeric mixture in quantitative yield.⁴



I

III. Collaborative Studies

In conjunction with R. Izac, of Project 2525, the monomethyl ester of a mixture of meso and *d,l*-2,3-dimethylsuccinic acids was prepared.⁵ Separation of the isomers by HPLC is under study.⁶ Derivatives of the acid have also been prepared.⁵

References;

1. H. Secor, 7566
2. D. Howe, 7575
3. C. Chaydarian, 7594
4. R. Southwick, 7446
5. L. Clawson, 7659
6. R. Izac, Project 2525

C. J. Chedman

2022151465

CHARGE NUMBER: 2501
PROJECT TITLE: Nuclear and Radiochemistry of Smoke
PROJECT LEADER: Roger A. Comes
PERIOD COVERED: August 1-31, 1981
DATE OF REPORT: September 9, 1981

^{14}C -Menthol Study¹

Samples of the cigarettes spiked with $^{14}\text{C}(\text{U})$ -menthol on 7/17/81 have been sectioned, extracted, and counted to determine if the menthol has equilibrated throughout the rod and filter. The cigarettes stored at 70°F and 65% RH had not reached equilibrium after 32 days. A single cigarette was placed in a 38°C oven for 5 days. Only a slight improvement was noted in the overall cigarette specific activity and a loss of some menthol is suspected under these conditions. The cigarettes will, therefore, continue to be stored under normal conditions until equilibrium is reached.

Harvey Oxidizer^{2,3,4}

The problems encountered with the Oxidizer have apparently been solved. Two approaches have led to satisfactory results being obtained. A return to the original liquid scintillation cocktail as supplied by the manufacturer has, for unexplained reasons, given acceptable data. In addition, a modification to the instrument has been made which allows the placement of the sample in a "cool zone" outside the combustion zone until O_2 flow is re-established. Both of these techniques are under further investigation to determine the best approach to finally settle on.

Low Level Laboratory⁵

Considerable progress has been made on the construction of the LLL. Exterior and interior block work is about 50% complete. The slab has been poured, the utilities tunnel to the power plant is nearing completion, drain pipes have been laid and electrical conduit and wall fixtures are being installed.

In conjunction with the construction, various building materials have been evaluated for their natural radioactivity levels. Total alpha plus beta activities ranging from 0.81 pCi/g for a "River Bed Aggregate" to 15.65 pCi/g for a "Light Granite" sample have been found. A sample of lightweight block was rejected based on its high beta activity. Samples of the mix used to pour the slab are currently under investigation. Monitoring of all building materials will continue.

2022151466

Cooked Flavors⁶

Ether extracts of 17 samples of cooked flavors (supplied by Charge Number 2305) were analyzed by capillary gas chromatography and GC-MS. The flavor samples were previously evaluated subjectively by application to and subsequent smoking of cigarettes. The samples were thus graded as to their relative acceptability. An analytical method was needed to verify this subjective evaluation and to allow future prediction for QA purposes. Results of the above GC analyses indicated that this analytical procedure or a variant of it might provide such a verification based on the amounts of certain organic acids present in the samples. Results and methodology were communicated to members of Charge Number 2305 and the development of the exact method turned over to the Analytical Research Division (Charge Number 1756).

Miscellaneous^{1,5,7,8}

1. Two lectures were given to personnel of the Analytical Division. The first was largely devoted to a discussion of the theoretical background related to radiochemistry and radiochemical measurements. The second involved health physics and laboratory procedures for handling radioactive materials with particular emphasis on carbon-14. The personnel attending were those who may be involved in various chemical analyses to be performed on carbon-14 and other radioactive materials.
2. A Special Report (Acc. #81-207), "WS-14 (N-t-butyl-p-menthane-3-carboxamide) as an Additive to Cigarette Paper", by A. F. Frisch and R. A. Kornfeld was issued.
3. A paper, "Preparation of ¹⁴C-Labelled Poly (ℓ-Menthyl Isopropenyl Carbonates)" by R. A. Comes and H. J. Grubbs was published in the Journal of Labelled Compounds and Radiopharmaceuticals, Vol. XVIII, No. 6, 769 (1981).
4. A course, "Environmental Occupational Radiation Protection," at the Harvard School of Public Health was attended by A. Frisch the week of August 24-28.
5. A new tissue solubilizer has been obtained (Solune®-350) and is under evaluation for use in determining specific activities on leaf and filler samples. To date it appears that digestion of tobacco materials is possible in a much shorter period of time than previously possible. Further studies are underway with various materials.
6. The GLRC (micro set-up) has been modified to accept a glass liner in the injection port. A 50m methyl-silicone capillary column was installed; a pressure regulated split-splitless injection system was fabricated and installed. Column efficiency has been optimized using injections of corn-mint oil and counting efficiency established at 3-4% with ¹⁴C-toluene.

2022151467

Various ^{14}C -labelled materials will be run to determine the versatility of the present system.

7. Optimization of counting techniques for ^{45}Ca containing materials continues. In addition, $^{45}\text{CaCl}_2$ has been applied to cigarettes and total smoke distribution studies are underway.

John Alon

References

- | | | |
|----|------------|---------|
| 1. | M. Chavis | NB 7615 |
| 2. | K. Barlow | NB 7547 |
| 3. | B. Francis | NB 7486 |
| 4. | R. Newman | NB 4818 |
| 5. | A. Frisch | NB 7309 |
| 6. | D. Douglas | NB 7064 |
| 7. | F. Greene | NB 7336 |
| 8. | G. Segura | NB 7502 |

2022151468

CHARGE NO: 2506
PROJECT TITLE: Botanical Investigations
PROJECT LEADER: Roger T. Bass
PERIOD COVERED: August 1-31, 1981
DATE OF REPORT: September 8, 1981

I. General

A. Upon completion of the harvest of the ^{14}C tobacco plants from run seven, all leaves were oven cured resulting in the following dry weight yields.

<u>Plant No.</u>	<u>Dry Wt.</u>
1 (N.rustica-G)	15 gm.
2 (Ox. 1-181)	106
3 (N.rustica-B)	32
4 (S.C.58)	113

Determinations for yield, total alkaloid, and specific activity showed plant #4, the S.C.58 to be the probable best selection from the four plant candidates grown in chamber run seven. Chamber run number eight with four plants of S.C. 58 was started on 8/5/81 and is currently in progress.

B. In cooperation with Charge #1716 the hydroponic culture experiment for feeding ^{45}Ca has been set up. A total of eight plants of Coker 319 has been arranged in a special area of the Greenhouse as directed by the Radiation Safety Officer. Two of the eight plants will be controls, four of them will be fed increments of ^{45}Ca weekly, and two plants will be fed ^{45}Ca in one total dose at the beginning of the experiment. Each plant is being grown in a 5 gal. container of nutrient solution with proper aeration. These plants have been under culture for about six weeks and are showing good growth. All plants have been topped and suckered during this period. Green leaf samples are being taken periodically for use by Charge No. 1716.

C. A list of the botanical names of natural ingredient plant sources of PM products was provided as requested by Dr. Wakeham.

D. A 10 gm. sample of Cambridge filler was prepared as requested by Charge No. 2501. The Kontes Chromaflex Sprayer was used to apply a 27 μCi water solution of $^{45}\text{CaCl}_2$ to the tobacco filler. After equilibration, a total of nine 85mm hand made cigarettes were prepared for smoke studies.

2022151469

II. Cooperative Studies

A. The 1981 Georgia Variety Evaluation display was held at Moultrie on September 26 & 27. For the Farm Test the seven entries (PDII, Coker 79-176MM, McNair 926, McNair 9107, N.C. TG-22, N.C. 85, and Va. 85) plus the two control varieties N.C. 2326 and N.C. 95 from the Hinson and Holton farms were evaluated. An average of about 76% of the tobacco was found to be suitable for PM use by Leaf Department personnel. The McNair 926, N.C. TG-22, and PDII graded highest based on physical appearance characteristics. For the Regional Small Plot Test, a total of 37 entries were evaluated with the Va. 85, N.C. 86, N.C. 69, and Speight G-86M showing up best in appearance.

B. A group of ground, dried Burley tobacco breeding line samples for 1981 was received from Coker's Pedigreed Seed Co. for total alkaloid analysis.

III. Greenhouse

A. The Kool-Cel evaporative cooling system was shut down in order to drain the water reservoir tank. Once empty, the tank was scrubbed and cleaned, and then refilled with fresh water in order to minimize the accumulation of foreign matter on the pads.

B. The normal plant production and maintenance activities have been completed. Trays of Coker 319, *N. sylvestris*, *N. tomentosiformis*, Coker 411, Ky. 14, *N. longiflora*, *N. glauca*, *N. glutinosa*, T.I. 1112, S.C. 58, and Speight G-28 have been seeded. Seedlings of Smyrna, Ky. 10, *N. rustica*, Coker 411, S.C. 58, Oxford 1-181, and Coker 319 were transplanted.

C. Fresh, green and cured tobacco plant materials were provided as requested for Charge Nos. 1716, 1901, 2501, and 1504.

IV. References:

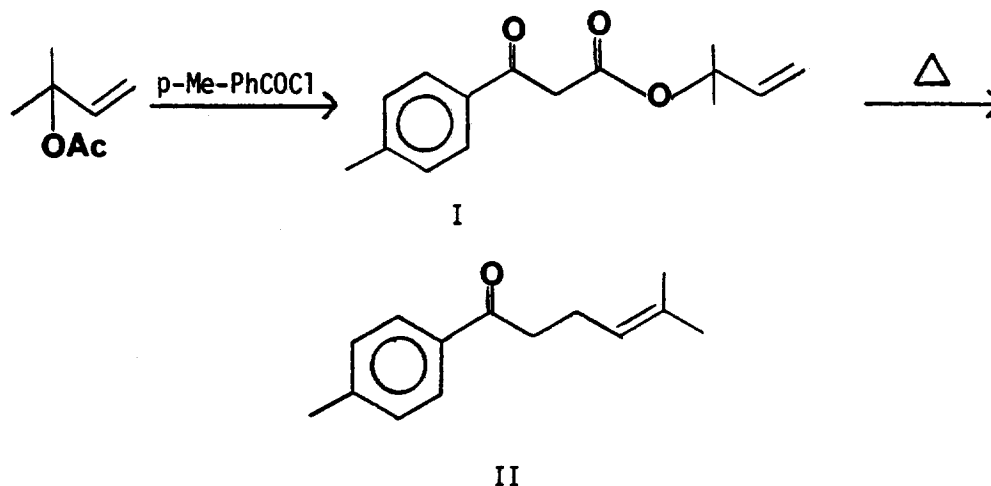
G. Newell NB 7395
A. Brna NB 7435

Roger T. Bass

2022151470

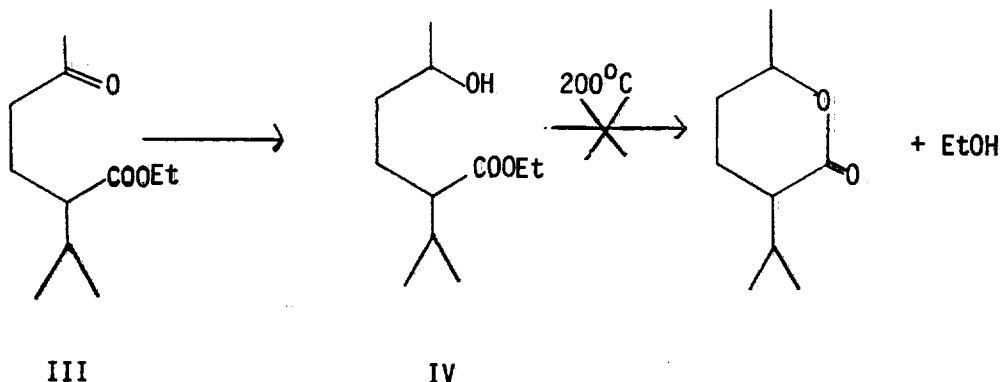
PROJECT NUMBER: 2515
PROJECT TITLE: Flavor-Release Chemistry
PROJECT LEADER: Y. Houminer
PERIOD COVERED: August 1-31, 1981
DATE OF REPORT: September 8, 1981

Last month we reported the discovery of an unusual rearrangement-decarboxylation of allylic β -ketoesters to generate homoallylic ketones. To test the generality of this reaction, 2-methyl-3-buten-2-yl acetate was prepared and its anion was reacted with p-methylbenzoyl chloride to give the desired keto-ester(I).¹ Pyrolysis of the latter at 200°C for 15 min. gave 71% of the rearranged ketone (II), thus demonstrating the generality of this reaction.¹ To the best of our knowledge this reaction which resembles the ester enolate Claisen rearrangement has not yet been reported in the literature. It has a great synthetic potential and we plan to study it with different allylic functional groups.

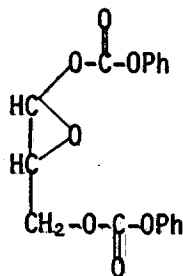


We have started to explore the release of alcohols and phenols from systems derived from esters of 4-hydroxyvaleric acid. Ethyl 2-isopropyl-5-oxo-hexanoate (III) was reduced with NaBH_4 to yield the corresponding hydroxy-ester (IV).² Pyrolysis of the latter at 200°C for 10 min. did not release ethanol and the starting material was left unchanged.² It is expected however, that more reactive esters such as phenyl esters will undergo this reaction at relatively low temperatures.

2022151471



Our research in the area of glycerol-based release systems has been extended to include also glyceraldehyde. In an attempt to peracylate the hydroxy groups with phenyl chloroformate an unexpected product was obtained (V).³ This very interesting bis-carbonate will be tested as a potential release system for phenols.



Y

Encouraged by the good yield of phenol released in the pyrolysis of 1-0-phenylcellobioside we extended this study to 1-0-(2-methoxy-4-methylphenyl)cellobioside. 1-0-(2-methoxy-4-methylphenyl)-2,3,6,2',3',4',6'-heptacetyl cellobiose was prepared in 65% yield from the octoacetate.¹ Removal of the acetyl protecting groups is now in progress.

The reaction of 2-ethylpyrazine anion with benzaldehyde was repeated to yield 15% of the expected product 2-(2-hydroxy-1-methyl-2-phenylethyl)pyrazine as a 1:1 mixture of diastereoisomers. The two isomers were separated by preparative t.l.c.³ Additional purification of these isomers is required before their pyrolysis can be studied.

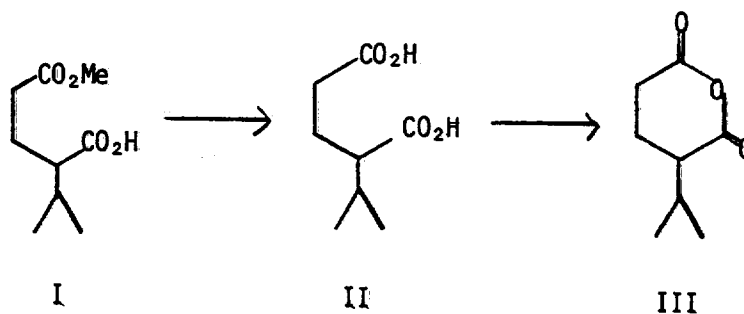
References

1. G. Chan 7462
2. Y. Houminer 7424
3. K. Podraza 7591

CHARGE NUMBER: 2520
PROJECT TITLE: Synthesis of Flavorants
PROJECT LEADER: William B. Edwards, III
PERIOD COVERED: August 1-31, 1981
DATE OF REPORT: September 9, 1981

I. Terpenoid Flavorants¹

Saponification of 4-carbomethoxy-2-isopropylbutanoic acid (I) gave the tobacco identical 2-isopropylpentanedioic acid (II). The acid (II) has a cheesy, valeric acid odor. Purification of II by gas chromatography is not possible, as the diacid dehydrates to the anhydride (III) under GC conditions. A sample of III will be prepared for flavor evaluation.

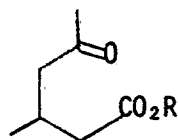


A mixture of methyl cis-4-isopropyl-7-methyl-5,7-octadienoate (IV, 20%) and methyl trans-4-isopropyl-7-methyl-5,7-octadienoate (V, 80%) was prepared from methyl 4-isopropyl-5-oxopentanoate via Wittig reaction with methallylidenetriphenylphosphorane. Following separation of the isomers, V will be hydrolyzed to the tobacco identical acid.



2022151473

With the purification of the esters (VIa-d) and the synthesis/purification of the ester (VIe), our work for a patent disclosure on long chain esters of oxoacids is completed.



- R=
- a) $-\text{CH}_2(\text{CH}_2)_8\text{CH}_3$
 - b) $-\text{CH}(\text{CH}_2)_5\text{CH}_3$
|
 $\text{CH}_2\text{CH}_2\text{CH}_3$
 - c) $-\text{CHCH}_3$
|
 $\text{CH}_2(\text{CH}_2)_{12}\text{CH}_3$
 - d) $-\text{CH}_2(\text{CH}_2)_{20}\text{CH}_3$
 - e) $-\text{CH}_2(\text{CH}_2)_7\text{CH}=\text{CH}(\text{CH}_2)_7\text{CH}_3$

1) D. Williams: N.B. 7618

William B. Elmer

2022151474

CHARGE NUMBER: 2525
PROJECT TITLE: Chemistry and Isolation of Tobacco Constituents
PROJECT LEADER: H. J. Grubbs
PERIOD COVERED: August 1-31, 1981
DATE OF REPORT: September 11, 1981

An additional harvest of yellowed Kudya leaves (*Pueraria labota*) has been carried out.¹ Using the methods previously reported, a steam distillation followed by methylene chloride extraction of the distillate has resulted in an essential oil. This essential oil was examined by capillary gas chromatography² and shown to be quite different from the essential oil of the green leaf. A number of tobacco identical flavorants including damasceone were identified. Harvest of additional leaf material will continue throughout the growth season with isolation and characterization of the essential oil.³

It has been well established that a number of amino acid-sugar compounds do occur naturally in bright tobacco.⁴ The isolation and quantitation of these compounds as a function of the natural ageing of flue-cured leaf tobaccos has been studied extensively.⁵ The content of 1-deoxy-1-proline fructose, the most prominent component of the amino acid-sugar compounds, increased during the initial period of storage. Only after two years of ageing did the level begin to decline.

The preparation and isolation of this tobacco identical Amadori compound is complete.⁶ Complete chromatographic and spectral characterization of this compound is currently under investigation. Concurrently, the preparation of the corresponding asparagine and valine tobacco identical Amadori compounds is underway. Pyrolysis studies with these compounds will aid in developing an understanding of their contribution to tobacco flavor and aroma.

At the request of Flavor Development, a seventy gram sample of highly purified WS-14 has been prepared and delivered for use as a reference standard. This material is currently being used to establish rigid specifications for purchase agreements with potential commercial suppliers of WS-14.⁷

In a collaborative effort with J. S. Long and Project 1101, we have initiated an isolation of azadirachtin.⁸ Two kilos of neem needs (*Azadirachta indica*) have been worked up in a new isolation scheme.⁹ Grinding of seeds in solvent, lipid removal, and column chromatography has resulted in a fraction of crude azadirachtin. Spectroscopic and chromatographic examination of this material is currently in progress. Purified samples of this fraction will be supplied to J. S. Long for assay as an insect antifeedant.

2022151475

Several new methods of isolation and quantitation of tobacco alkaloids have been developed. In conjunction with Project 2506, the alkaloid content and alkaloid distribution (mass and radiochemical) has been determined for the four different cultivars of $^{14}\text{CO}_2$ chamber grown tobaccos.¹⁰ With the arrival of one additional minor alkaloid standard, the quantitative data will be available for publication. This procedure, as well as an auxillary HPLC procedure, is currently being developed as a preparative method.¹¹

References;

1. R. Southwick
2. R. Hale
3. With R. Southwick in a collaborative effort.
4. M. Noguchi, et. al.; Agric. Biol. Chem., 35(1), 65-70, 1971.
5. M. Noguchi, et. al.; ibid; H. Shigematsu; Agric. Biol. Chem., 41(12), 2377-2385, 1977.
6. S. Haut
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9. R. Izac
10. M. Edmonds
11. J. Brady, R. Izac, M. Edmonds

Harry Crabbe

2022151476

CHARGE NUMBER: 4009
PROJECT TITLE: Smoke Studies
PROJECT LEADER: B. Goodman
PERIOD COVERED: August, 1981

I. HUMAN SMOKER SIMULATOR

A. Extended Smoking Study

All panelists are in the stage of being recorded again on the regular brand they smoked prior to switching to Merit Ultra Lights. The collection of profile data will be completed in September.

B. Simulator Equipment

A digital flow meter was tested for flow adjustment with the result that a new valve system was found to be needed. The necessary valves to obtain the desired maximum flow have been ordered.

Training in usage of the microprocessor system is progressing smoothly. Some minor changes are being made to handle exceptional cases before the system becomes established in September. At that time the present system in the computer room will be terminated.

II. WRAPPER AND FILLER MODIFICATIONS

A. Base Paper Study

Handsheets containing titanium dioxide as the whitening agent were made in sufficient quantities to splice into a small bobbin. A trial machine run will be made with this paper to determine the feasibility of using hand-spliced bobbins for other applications.

The data collected on handsheets made with CaCO_3 filler, including porosity, thickness, percent calcium and basis weight, were analyzed in view of the independent variables of weight of fiber, weight of filler, volume of stock and drain time. Using a combination of graphs and computer programs, a method was developed for producing a handsheet having a desired basis weight and calcium content and a predicted porosity.

B. Low Ash Wrapper

A cigarette wrapper with 4% chalk, prepared by Schweitzer Paper Company, has been tested for gas phase deliveries. Some reductions on a per puff basis were found relative to a full chalk, non-additive wrapper. Total puff count increased from nine puffs on the control to twelve puffs on the low ash wrapper. To lower the puff count, various burn promoters will be applied in-house to a low ash wrapper.

2022151477

C. Cation/Anion Ratio Study

A second series of wrappers has been prepared and tested for varying the cation/anion ratio of sodium citrate additions. Analysis of delivery data did not confirm previous trends to lower CO delivery as citrate increases at constant sodium levels. Another run is planned using the same wrappers to evaluate both sets together. Analysis shows paper burn rate to be useless in predicting SBT, puff count, or any delivery data.

Regression analysis to eliminate the effects of tobacco weight, O.V. and circumference using the APL modeling package will begin in September.



B. Goodman

BG/lad

2022151478

CHARGE NUMBER: 4010
PROJECT TITLE: Brand Modifications
PROJECT LEADER: W. A. Geiszler
PERIOD COVERED: August, 1981

I. TAR/CO DELIVERY CHANGES

A. Marlboro (L. Stewart)

A POL test is in progress comparing a Marlboro KS model with a 14 mg FTC tar delivery against the 16 mg production control. Preliminary results indicate that the experimental cigarette is achieving parity in preference with the control. The experimental cigarette uses a modified flavor formulation. Similar POL testing is planned for 14 mg LS and 100 mm models.

An HTI test is scheduled to compare a 16 mg FTC tar/14 mg CO Marlboro KS model against the 16 mg tar/16 mg CO production control.

B. B&H 100's (L. Stewart)

Model making is continuing to develop B&H 100's prototypes which deliver 16 mg FTC tar and 14 mg of CO for consumer panel testing against the 16 mg tar/17 mg CO production control.

C. Marlboro Lights (B. Mait, J. Nepomuceno)

An HTI test is in the field comparing a 9 mg FTC tar version of Marlboro Lights 100 against the 11 mg production control. A Marlboro Lights 100 model with "equal tar/reduced CO" relative to the production control is being evaluated by in-house panels.

D. B&H Lights (B. Mait)

A B&H Lights model with a reduced CO delivery is being tested against the production control by in-house panelists. Both the experimental and control cigarettes have the same FTC tar delivery.

E. Merit (B. Mait, S. Stone)

A POL test is in the field comparing a Merit KS model with reduced CO delivery against the production control cigarette, both having the same FTC tar delivery. Another reduced CO delivery Merit KS model has been made with porous tipping and is being tested by in-house panelists.

F. Virginia Slims Lights (L. Stewart)

A program to reduce the CO delivery of Virginia Slims Lights by 1 - 2 mg is in the model making stage.

2022151479

G. Cambridge (S. Stone)

Models of Cambridge KS and 100's designed to deliver 3 and 5 mg of FTC tar respectively are undergoing smoking analysis.

II. CIGARETTE TECHNOLOGY

A. Hot Melt Tipping Adhesive (J. Nepomuceno)

Cigarettes were made for a POL test to evaluate an Ecusta hot melt tipping adhesive versus the production PVA adhesive on Marlboro KS cigarettes. Both cigarettes met analytical smoking specifications, but the experimental cigarette was judged to be significantly different from the control by both the Flavor Panel and the MC Panel. More cigarettes are being made with different batches of Ecusta hot melt tipping to identify the source of the taste problem.

B. Porous Plugwrap (B. Mait)

Schweitzer high porosity plugwraps with CORESTA values of 15,000 and 19,000 are being evaluated as replacements for 260-M1 plugwrap to achieve cost savings. Cigarette testing at R&D indicates that the 19,000 material is the better replacement candidate. Small scale factory trials are scheduled for both materials on the Cambridge 100 mm product.

An evaluation of Ecusta 28070 plugwrap has been completed which shows the material to be very comparable to Schweitzer's 65-M1 product. Since 65-M1 is not yet being used in production, no factory testing of 28070 is planned at this time.

C. Cork Tipping (S. Stone)

An Ecusta cork tipping colored with Fe_2O_3 in place of azo dyes is being evaluated for electrical perforation. The perforating performance of the experimental tipping was satisfactory in a 10 bobbin trial, but the perforations are slightly visible on the cigarette. Further testing is being conducted to minimize the visibility of the perforations.

W. A. Geiszler

W. A. Geiszler

WAG/lad

2022151480

CHARGE NUMBER: 6906
PROJECT TITLE: BIOLOGICAL EFFECTS OF SMOKE
PERIOD COVERED: August 1-31, 1981
PROJECT LEADER: R. A. Pages
WRITTEN BY: T. Yu
DATE OF REPORT: September 8, 1981

1. E. COLI (λ) PROPHAGE INDUCTION ASSAY¹

A systematic evaluation on the prophage inducing activity of CSC base fraction samples derived from 14 cigarette types were performed in the plate test. The experiments were designed to determine the minimal inducing concentration (MIC) of each sample in order to separate the samples on the basis of activity. The results showed that the samples can be separated into four groups: 1) No detectable activity: low tar reference, LTF-3A, 2A1, and ET. 2) The MIC of 2R1, bright + NO₃ and RCB was 0.1 μ g. 3) The MIC of ERKS, burley, DIET, RKS, and bright was 1 μ g. 4) the MIC of LTF-2A was 25 μ g.

2. V79 CHINESE HAMSTER CELL MUTATION ASSAY^{2,3}

Cells were obtained from an outside laboratory. Frozen stock cultures have been made in our laboratory for future use. Currently, the growth characteristics of the cells are being studied.

3. YEAST MITOTIC GENE CONVERSION ASSAY⁴

A good working stock culture of strain D₄ was obtained using the procedure recommended by an outside laboratory.¹³ Samples (prepared by Project 6908 personnel) of mainstream smoke (MS) and sidestream smoke (SS) derived from 2R1, bright, burley, RCB, and RL reference were tested using the new stock culture. These samples consisted of materials collected in impaction traps and on Cambridge pads. In addition, the SS samples were generated and collected under both static burning (SB) and static/dynamic burning (S/D) conditions.⁵ The experimental data are being analyzed, particularly with respect to water content, which ranged from 8 to 80% in these samples. SS samples contained much higher water content than MS samples. The results and additional experiments of this work will be reported next month.

A culture of *S. cerevisiae* strain D₇ has been added to our culture collection. This strain is suitable for detecting multiple endpoints of genetic effects including mitotic gene conversion and recombination, point mutation and reverse mutation. Experiments are planned to compare the sensitivity of strains D₇ and D₄.

2022151481

4. SALMONELLA/MICROSOME MUTATION ASSAYA. Pyrolysis Studies (with 6908)⁶

The specific activities of burley and bright pyrolysates were determined in TA98 in the presence of microsomes. Pyrolysis was carried out in nitrogen from 400°C to 800°C at 100°C intervals for 12 min. Burley pyrolysate showed a higher level of activity (about two to five fold) than the bright samples. The increase in the specific activity as a function of temperature was similar in both samples, except for the 800°C sample. Burley pyrolyzed at 800°C showed a pronounced increase in the specific activity; whereas the specific activity of bright leveled off at 700°C. More work will be done to substantiate these observations.

B. CSC Base Fraction Activity (with 6908)⁶

The pot residue of a base fraction of burley CSC was partitioned into toluene and 50% methanol. The toluene fraction was subsequently chromatographed on a LH-20 column. It was found that 65-70% of the total activity was located in three of the ten fractions collected. These three fractions represented about 5% of the total weight.

C. Mainstream Smoke (MS) versus Sidestream Smoke (SS) Activity (with 6908)⁷

Samples tested and sample preparations were the same as those described under item 3 of this report. The specific activities of these samples were determined in TA98 plus microsomes. The results showed that MS samples were more active than SS samples. These results are in agreement with previous observations.⁸ The rank order of activity is different in MS and SS samples collected under either SB or S/D burning conditions. For the MS IT CSC, the order of specific activity was: burley>RCB>2R1>RL reference>bright. This rank order is similar to results obtained in the past.⁹ The specific activities of MS TPM of the five cigarette types studied were either similar to that of MS IT CSC (*i. e.*, RCB) or lower (*i. e.*, burley, and RL reference). For the SS samples, the specific activities of burley, bright, and RL reference collected under S/D burning conditions were higher than those collected under SB conditions. For RCB and 2R1, the SS samples showed comparable levels of activity regardless of the burning conditions.

D. Water Expanded Burley Tobacco⁷

CSC samples, prepared from cigarettes which contained water expanded and control, cased and uncased, burley tobacco (furnished by H. Sun) were tested in TA98 plus microsomes. The results (along with those of an analysis of variance conducted by J. E. Tindall), shown below, indicated that: 1) the cased samples were significantly less active than the corresponding uncased samples; and 2) the expanded samples were significantly less active than the control samples.

2022151482

<u>CSC</u>	<u>TA98 Activity</u> ^{a,b}
Uncased Control	4514 ± 227]
Uncased Expanded	3632 ± 558]
Cased Control	3012 ± 302]
Cased Expanded	2312 ± 336]

a) TA98 activity = rev/mg of CSC. The numbers represent the mean ± SD of determinations conducted on four separate CSC preparations from each cigarette type.

b) Samples not within the same bracket are different, $p < 0.05$.

E. Miscellaneous⁷

One sample was tested at the request of J. E. John.

5. L5178Y THYMIDINE KINASE MUTATION ASSAY^{10,11}

The cloning efficiency (CE) of L5178Y cells obtained from ten solvent control (DMSO) samples was $56 \pm 11\%$. This is an acceptable value according to the SOP established in our laboratories.¹² The dose response of 2-acetylaminofluorene was established in a separate experiment; the CE values obtained from the solvent and the positive control compound were all acceptable. It was therefore decided that assay work will now be resumed. For next month, the activity of three carbonyl compounds known to be present in large amounts in cigarette smoke will be investigated.

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Perry Yu

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CHARGE NUMBER: 6908
PROGRAM TITLE: SMOKE CONDENSATE STUDIES
PERIOD COVERED: August 1-31, 1981
PROJECT LEADER: R. N. Ferguson
DATE OF REPORT: September 8, 1981

A. SALT EFFECTS (with 6906)

The level of filler potassium was lower than expected¹ for the salt sprayed RL base web samples and the control which were prepared last month.² Rather than proceeding to base fraction studies as planned, other analytical data were evaluated and a few handmade cigarettes were prepared and smoked.^{1,3} The delivery of the KOAc sprayed base web cigarettes was half that of the control, while the KCl sprayed sample showed an intermediate delivery, in line with previous results. The activity of these IT CSCs are also being evaluated in the *Salmonella*/microsome assay before the larger scale base fraction preparation is conducted.

B. MW 288⁴

Study of the fractions isolated from the thermal decomposition products of α -4,8,13-duvatriene-1,3-diol continued.² A MW 288 aldehyde of unknown structure is present in one fraction. Further studies are planned to elucidate the structure of this aldehyde.

C. NITROSAMINES⁵

Acceptable gc column performance for the nonvolatile nitrosamine analysis was reestablished. A number of RL samples were analyzed for nonvolatile nitrosamines in filler and smoke. Work is also progressing on a survey of filler types for nitrosamine deliveries to smoke. Cigarettes have been obtained for an initial evaluation of dilution and filter effects on both volatile and nonvolatile nitrosamines in smoke.

D. PAH PROCEDURE⁶

Either EM Si-60 or Si-40 silica gel column chromatography was found to be satisfactory for the first step in the PAH isolation procedure. Florisil PR 60/100 will probably be used for the third step but some problems with selective PAH removal were encountered. It is expected that some additional optimization of the activation conditions and the elution solvent composition will give a satisfactory overall procedure.

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A Jennings bonded phase capillary gc column is being evaluated using the HP on column injector. Both the on column injector and the column have performed well for the PAH analysis.

E. CHROMATOGRAPHY AND GC/MS

A 600 mg sample of X6D3IM IT CSC was used to evaluate the potential applicability of the Chromatotron® system to smoke condensate fraction studies. Despite problems due to the complexity of CSC, this rapid preparative chromatographic procedure appears to be well suited to present and planned fractionation studies.⁷

A gc/ms analysis of air contaminants was conducted as part of a collaborative assistance to the Manchester Fire Department.^{8,9} Several fractions were also examined by gc/ms as part of our studies of carbolines in the base fraction of smoke. A major problem with these studies is the lack of components amenable to either packed or capillary gc.^{8,9} The availability of a "stand alone" Sigma 3 gc, as part of the overall new VG MM16 gc/ms system, should provide the opportunity for more developmental chromatographic studies. We expect to transfer the current du Pont 21-490 gc/ms/ds to Chemical Research and to install the new system during September.^{8,9}

F. ACTIVE BASE FRACTION COMPONENTS (with 6906)

A larger sample of the X6D3IM base fraction (50% methanol extract) was chromatographed on Sephadex LH-20. There were some small differences from the previous small scale experiment and from the three large scale separations of the toluene extract on LH-20.² However, the 50% methanol extract fractions continued to show enhanced accountability, indicating the potential activity of the parent fraction is diminished by some interactive effects among its components.¹⁰

Efforts to determine if the active carbolines, Trp-P-1 and Trp-P-2, are present at significant levels in CSC continued this month. These are produced from the pyrolysis of tryptophan, but the low level of this amino acid in filler made it difficult to establish if any Trp-P-1 or Trp-P-2 was present in X6D3IM CSC.²

A LTF-2A filler with added tryptophan (1% filler nitrogen) was selected as an optimal test case for Trp-P-1 and Trp-P-2 formation during smoking. Before beginning the experiment, the cleanup procedure was optimized using a mixture of Trp-P-1, Trp-P-2, harman, and norharman. The first step was silica gel column chromatography of the base fraction. The next step was tlc under carefully controlled conditions. Final cleanup used RP-HPLC with fluorescence detection. A 2.1 g sample of IT CSC was used for the actual experiment. Neither Trp-P-1 nor Trp-P-2 were detected after fractionation of this sample. Based on the detection limits of the

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procedure, Trp-P-1 and Trp-P-2 (if present at all) cannot make a significant contribution to CSC activity.¹¹ We plan to conclude this study in the near future based on these results.

Studies directed at determining if Glu-P-1 and Glu-P-2 (active pyrolysis products of glutamic acid) are present in burley base fraction continued.¹² The first step, as in the Trp-P-1 study, was silica gel chromatography of the base fraction. A silica gel TLC procedure was developed for the next step. For preparative TLC, CHCl₃/acetone (4/1) with multiple development of the plates, gave satisfactory results. The enriched fraction obtained in this manner is now being used for RP HPLC profiling.

G. PYROLYSIS (with 6906)^{3,13}

The evaluation of the activity of tobacco pyrolyzates continued with a study of bright and burley fillers heated in nitrogen. Samples were prepared over the 400° to 800°C range at 100° increments. As usual, data related to weight loss and pyrolyzate collection were obtained. The *Salmonella*/microsome assay results for these samples showed: the burley pyrolyzate was more active at all temperatures than the bright pyrolyzate; burley produced optimal activity at 500°, while bright produced optimal activity at 700°. These results will be reconfirmed and extended in studies now in progress. The first samples will explore the effects of He *versus* N₂ as pyrolysis atmosphere and the effect of extensive purging of the filler with either gas prior to pyrolysis.

H. SIDESTREAM SMOKE STUDIES (with 6906)³

A series of sidestream smoke samples were prepared from Kentucky reference, RCB, burley, bright, and RL reference cigarettes. Included in the evaluation were various trapping procedures, static *versus* dynamic burn, and mainstream samples for reference. The assay results are reviewed in detail in the current 6906 Progress Report. This experiment provided the basis for more extensive studies which will use TPM since both MS and SS samples can be obtained from the same cigarettes during dynamic burn. Experiments are now in progress to evaluate the procedure to be used for TPM preparation for these studies.

I. BURLEY CSC FRACTIONATION (with 6906)¹

Experiments were designed to evaluate the activity of smoke fractions more completely in the yeast assay. Burley IT CSC was used for acids, bases, neutrals, and residual water solubles fractionation. In addition, a water-ethyl acetate partition was conducted. Further fractionation will depend on the results of the biological evaluation of these samples.

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